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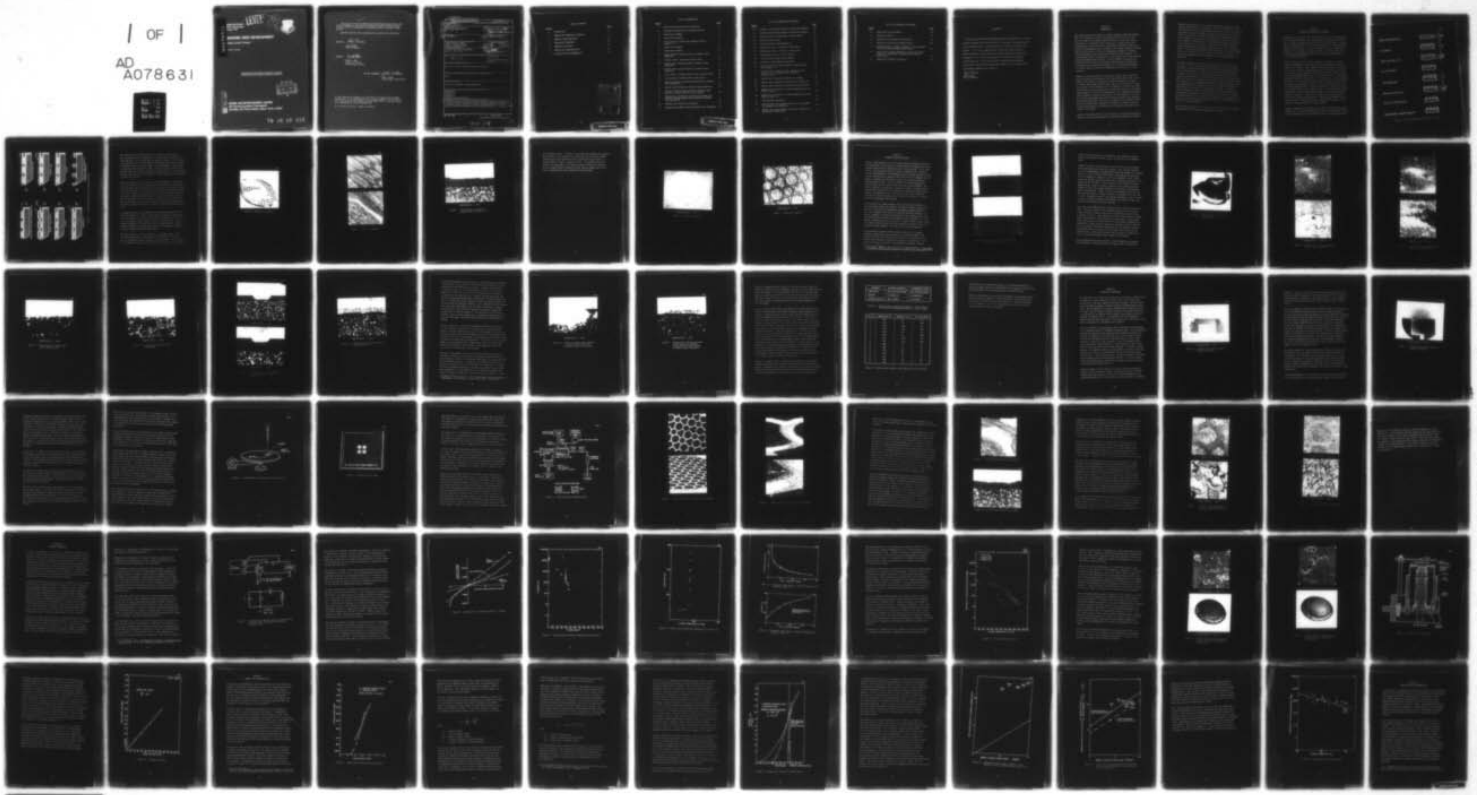

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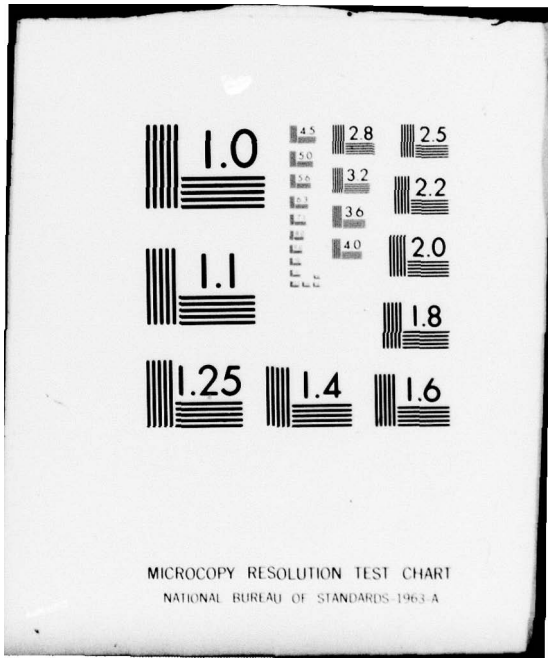
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LEVEL II

RADC-TR-79-249
Final Technical Report
October 1979

BONDED GRID DEVELOPMENT

Hughes Aircraft Company

Richard Dawson

ROME AIR DEVELOPMENT CENTER
Air Force Systems Command
Griffiss Air Force Base, New York 13441

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER: RADC-TR-79-249	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle): BONDED GRID DEVELOPMENT	5. TYPE OF REPORT & PERIOD COVERED: Final Technical Report, 1 Feb 78 - 31 Jan 79
6. PERFORMING ORG. REPORT NUMBER: N/A	
7. AUTHOR(s): Richard Dawson	8. CONTRACT OR GRANT NUMBER(s): F30602-78-C-0101
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Hughes Aircraft Company, Electron Dynamics Division, 3100 W. Lomita Boulevard, Torrance CA 90509	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 62702F, 45061226
11. CONTROLLING OFFICE NAME AND ADDRESS: Rome Air Development Center (OCTP), Griffiss AFB NY 13441	12. REPORT DATE: Oct 79
	13. NUMBER OF PAGES: 85
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office): Same	15. SECURITY CLASS. (of this report): UNCLASSIFIED
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE: N/A

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

Same

18. SUPPLEMENTARY NOTES

RADC Project Engineer: Leon Stevens (OCTP)

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Bonded Grid
Hafnium Oxide
Microwave
Electron Gun
Thermionic Tube Control Grid

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report details the results of efforts expended towards the development of a cathode and control grid assembly having the control grid solidly bonded to the cathode surface by means of a layer of insulating material. A device capable of emission and modulation was fabricated and tested. → to pg. viii

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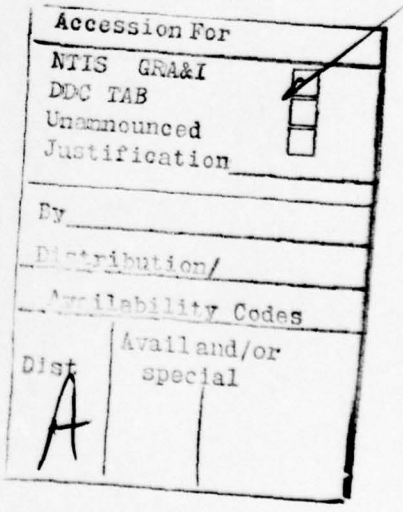
TABLE OF CONTENTS



LIST OF ILLUSTRATIONS

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EVALUATION

This effort under RADC TPO4B demonstrated the feasibility of a control grid for microwave tubes fabricated with lithographic techniques directly on the cathode surface. When perfected this technique has two primary payoffs: (1) It will permit the use of high gain control grids at higher frequencies than possible with present shadow grid construction techniques, (2) It will also improve tube reliability, yield and construction cost. This technique will be applicable to microwave tubes used in tactical and ground based radars as well as ground based and satellite communications systems.

Leon Stevens

LEON STEVENS
Project Engineer

SECTION 1
INTRODUCTION

This report details the results of efforts expended towards the development of a cathode and control grid assembly having the control grid solidly bonded to the cathode surface by means of a layer of insulating material. During the course of this work it became clear that attention must be concentrated on developing the process by which the insulator was produced; and, accordingly, the program ultimately focused upon the initiation of a series of experiments designed to optimize the coating process and to evaluate the material thus obtained. The remaining fabrication techniques were also refined; and in some instances revised approaches were explored. A device capable of emission and modulation was fabricated, potentially demonstrating the feasibility of manufacturing a bonded grid assembly, but considerable improvement is required before the practical advantages of such a device can be realized.

Samples of insulating material obtained by chemical vapor deposition during preliminary investigations had shown great promise; and, consequently, this program was originally intended primarily to resolve incidental fabrication problems, adapt the various techniques to spherical cathodes, and incorporate other refinements in the grid structure. Unfortunately, the poor adhesion and inconsistent quality of subsequent samples impaired this work and demonstrated that, although the chemical vapor deposition technique could satisfy the requirements of this program, further development and stricter process controls were necessary. An extensive experiment involving the preparation and coating of specimens was initiated and, although not finished, has already revealed several possibilities for improving the preliminary processing of the surface to be coated.

Despite frustration caused by the difficulty in obtaining satisfactory samples of the insulating material, substantial progress was realized

towards the development of the various techniques required to create the grid structure on a flat cathode. Plans for dealing with spherical geometries and grid encapsulation were postponed until the problems encountered in photolithography, mechanical polishing, and reactive plasma etching as well as chemical vapor deposition might be resolved for the simpler flat geometry. Grid patterns consisting of 0.0035" wide elements circumscribing 0.012" openings were produced with excellent resolution, but difficulty in obtaining the desired insulator thickness prevented the completion and testing of a fully functional device. Two very promising pieces were fabricated, one of which demonstrated the suitability of the insulator's dielectric properties and one of which demonstrated the feasibility of achieving emission and modulation.

The type of material to be used as an insulator was a key issue in this project. Both aluminum oxide and hafnium oxide were considered throughout the program, hafnium oxide offering the more promising balance of characteristics. Several devices were constructed in order to measure the dielectric properties of hafnium oxide as obtained by chemical vapor deposition. The resistivity and permitivity were determined as functions of temperature and compared with the published values for hafnium oxide. The greatest resistivity observed at typical operating temperatures was considerably less than the published value for bulk material, but adequate for the purpose at hand. A definitive value of permitivity was not obtained, but the values observed were substantially in agreement with those expected. Attempts to formulate an aluminum oxide layer by chemical vapor deposition were unsuccessful, and a layer of the material produced by sintering a paste proved detrimental to cathode performance.

The following sections of this report describe the concept being developed and the efforts expended towards that development.

SECTION 2

BONDED GRID FABRICATION TECHNIQUE

Ultimately it is intended that the bonded control grid be encapsulated with insulating material. This encapsulation is expected to be necessary in order to inhibit undesirable emission from the grid. Furthermore, if the bonded grid is to find wide application in modern, convergent flow electron guns, a means of forming the grid on a spherical cathode will be required. To date, however, the work has been aimed at creating an exposed bonded grid, that is without encapsulation, on a flat cathode surface.

Figure 1 shows the basic technique employed to produce the exposed bonded grid assembly. The porous tungsten cathode pellet is first coated with a layer of dense tungsten by means of chemical vapor deposition. This surface is then mechanically polished both to give precise control of the coating thickness and to provide a smooth surface for subsequent photographic work. The polished surface is next photomasked and etched to produce grooves corresponding to the grid elements. The piece is then coated with hafnium oxide by chemical vapor deposition which fills the grooves, but coats the remaining surface as well. The excess hafnium oxide is removed by polishing, leaving a grid insulator inlaid within a dense tungsten layer. A second reactive plasma etch removes the dense tungsten and exposes the cathode's emitting surface. Finally, a few microns of tungsten are sputtered across the surface to form a grid conductor. Experience has shown that this layer provides a sufficient conduction path without shorting to the cathode or impairing emission from the cathode.

Figure 2 illustrates the technique envisioned for producing an encapsulated grid. As with the exposed grid, the process begins by coating a porous tungsten pellet with dense tungsten to a precise thickness,

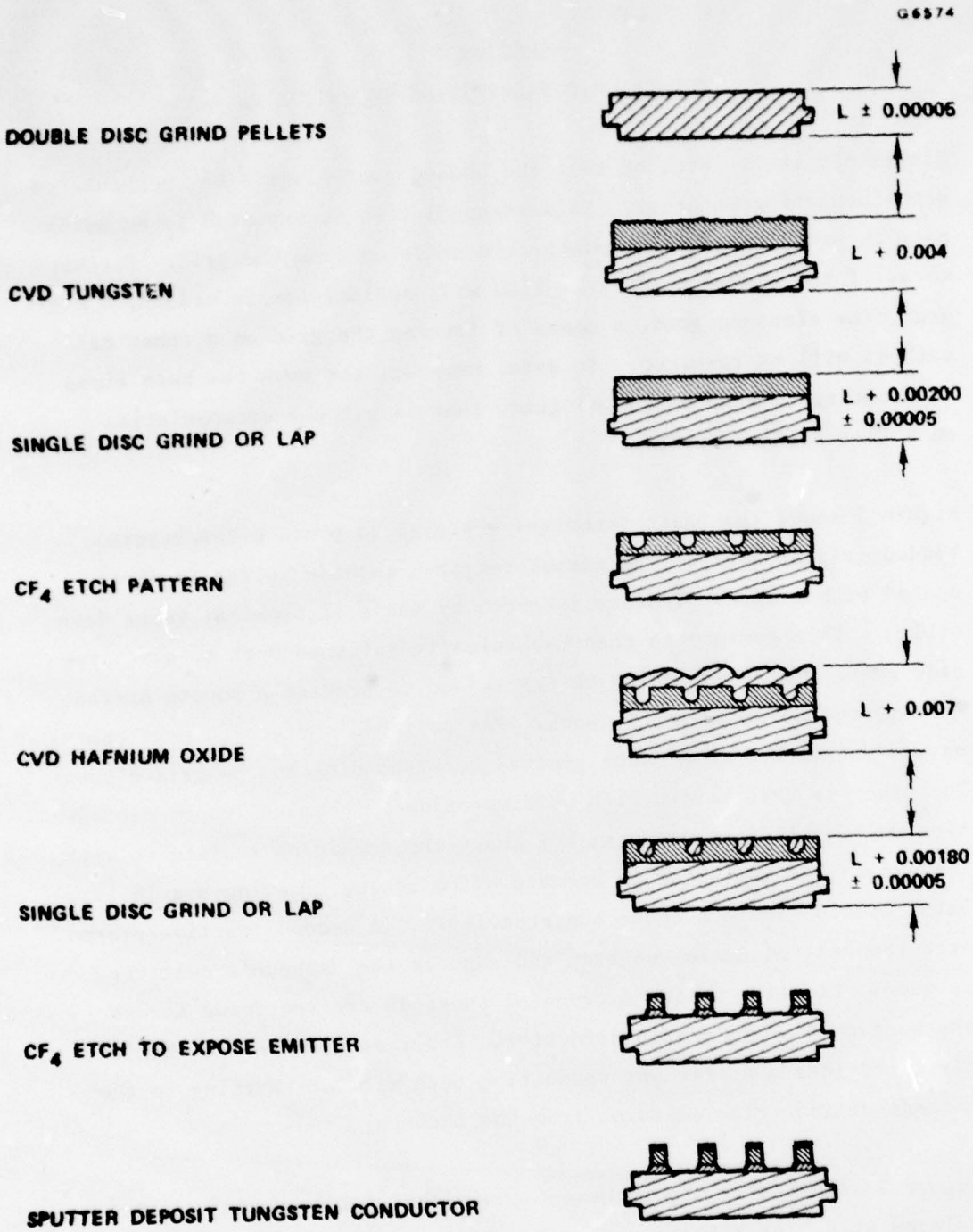

Figure 1 Basic bonded grid fabrication technique.

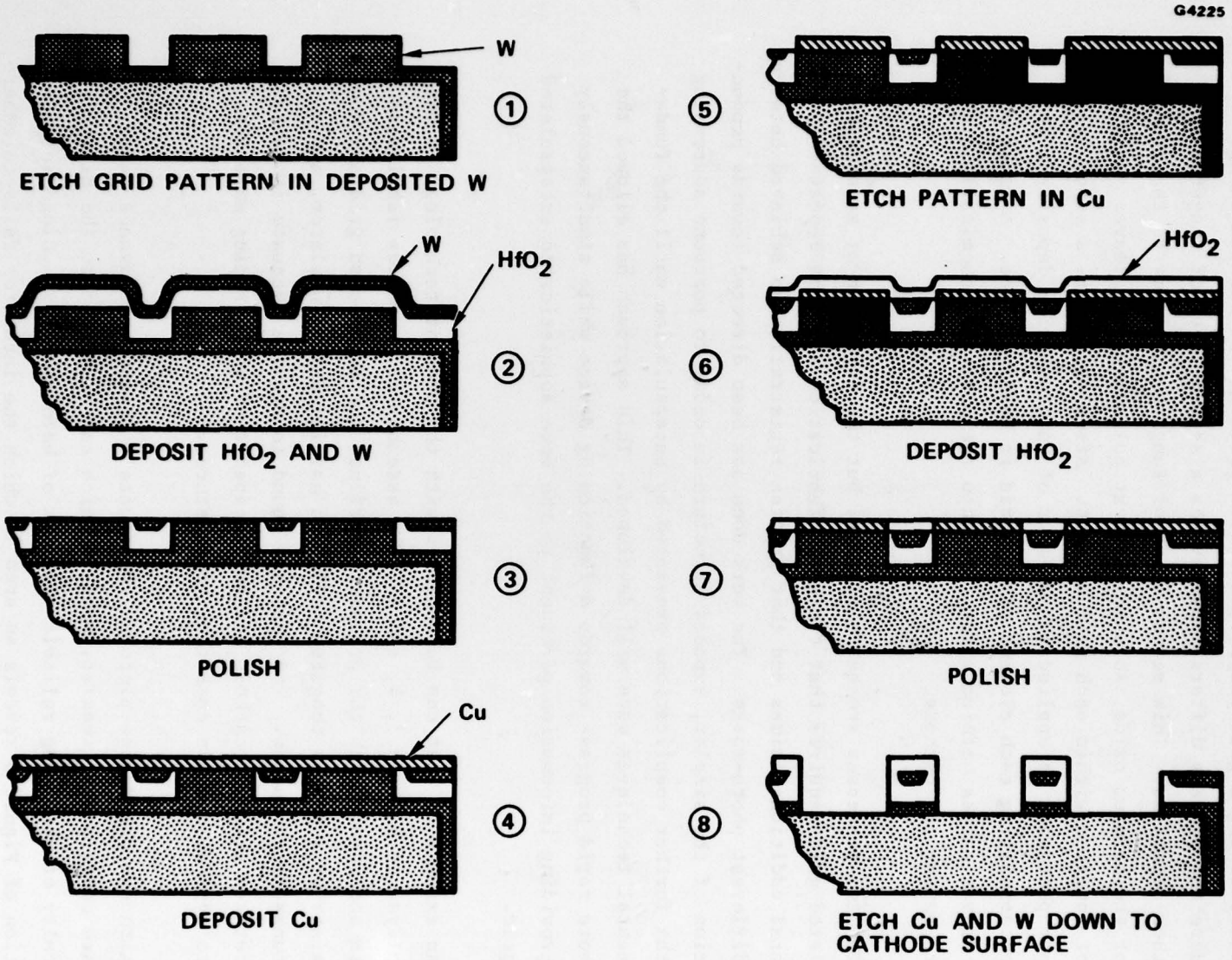

Figure 2 Proposed encapsulated grid formation process.

photoetching a grid pattern, and then coating with hafnium oxide. Here the process differs, however, as a second layer of tungsten is then deposited. This second layer of tungsten conforms to the contour of the hafnium oxide, so that the next polishing step leaves an island of tungsten within each grid element. After polishing, a second copper photomask is applied and a layer of hafnium oxide deposited, thus encapsulating each element of the grid in hafnium oxide. A final polish removes excess hafnium oxide, and then the parts are chemically etched to expose the cathode.

The two processes are quite similar, but the formation of an encapsulated grid requires that the basic fabrication steps be repeated several additional times and that precise registration be achieved between different photo-masks. The work done has been directed towards production of the simpler, exposed structure in order to postpone addressing the further complications presented by encapsulation until the fundamental techniques were well developed. This approach has allowed the more rapid progress towards a functioning device while simultaneously providing information pertinent to the more sophisticated encapsulated grid.

An example of what can be achieved with the present technology is displayed in Figures 3, 4, and 5. The assembly pictured was fabricated in accordance with the procedure outlined for an exposed grid and consists of a porous tungsten pellet, a hafnium oxide insulator, and a tungsten conductor. The unit was found to provide adequate isolation between grid and cathode and to be capable of withstanding exposure to typical cathode operating temperatures.

Although the device pictured represents a tremendous advance there are certain deficiencies, most of which can be traced to the difficulty of obtaining reliable coatings of hafnium oxide. Close inspection of Figure 3 reveals an area in which the insulator fails to adhere

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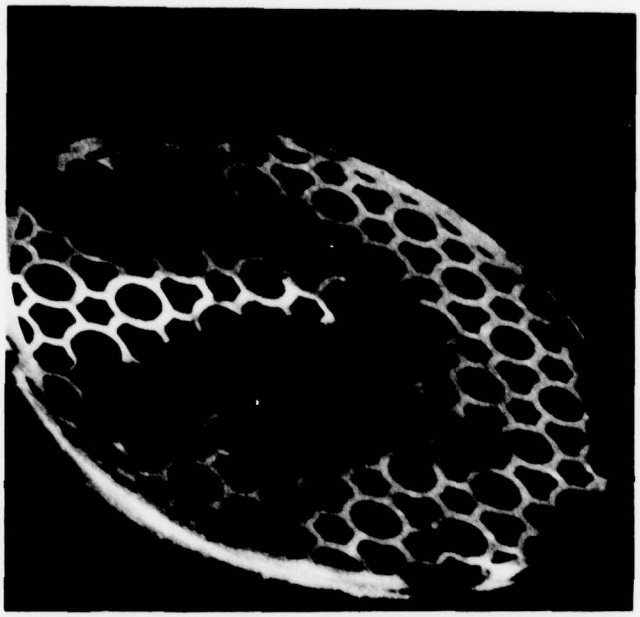

Figure 3 Bonded grid assembly.

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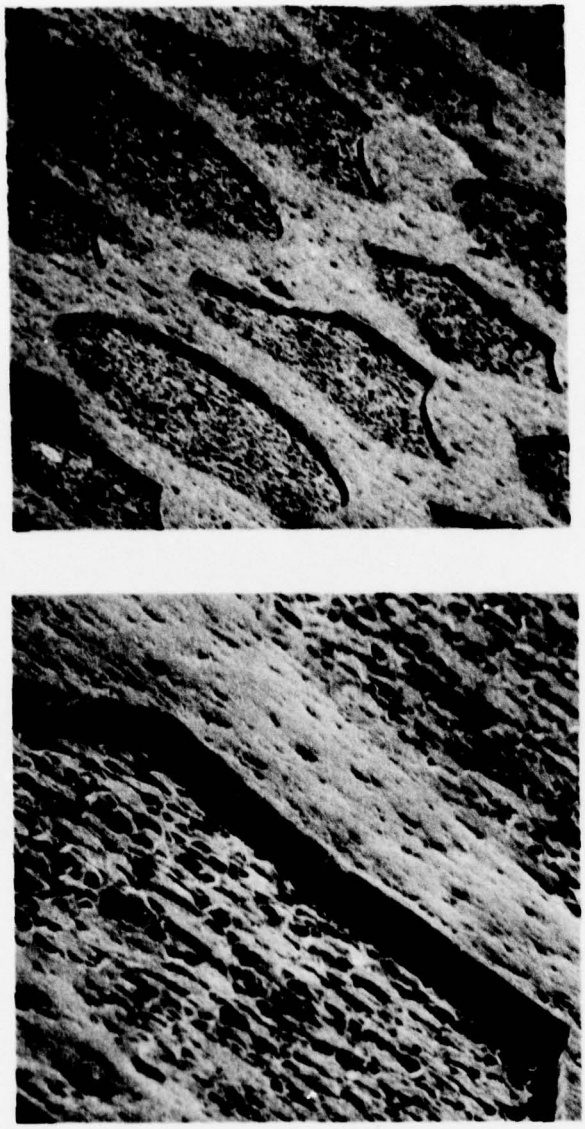

Figure 4 Bonded grid assembly.

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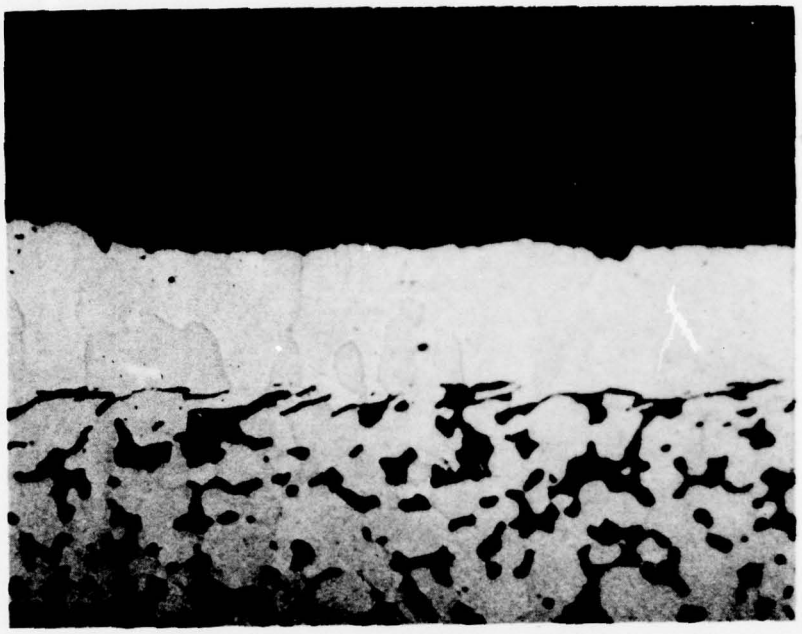

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Figure 5 Cross section of bonded grid assembly shown in Figures 3 & 4.

to the pellet surface. In Figure 5 two additional problems are apparent. The thickness of the insulator is insufficient and the dense tungsten coating has not been removed from the intended emitting surface. A second example is shown in Figures 6 and 7. This assembly was fabricated in the same manner, but superior adhesion between the insulator and the cathode surface allowed the final reactive plasma etch to proceed to completion and fully expose the emitting areas.

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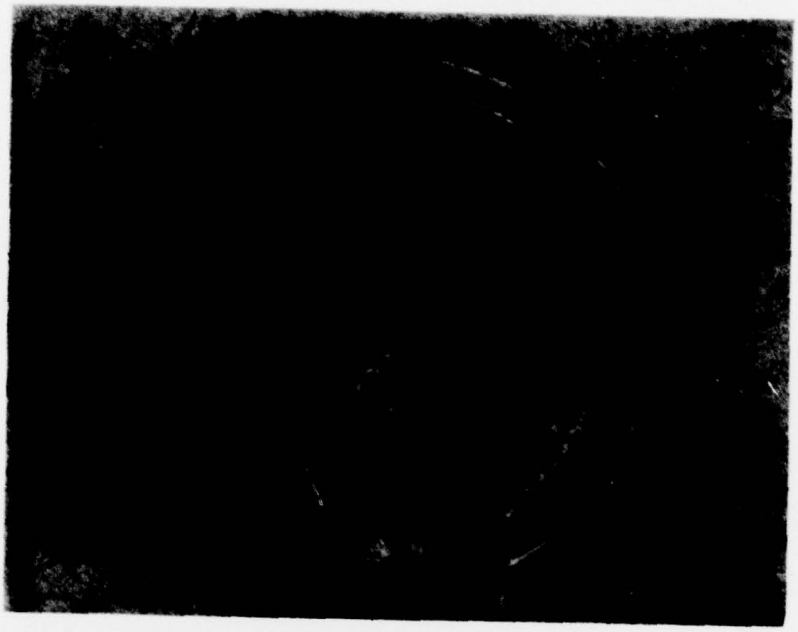

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Figure 6 Bonded grid assembly.

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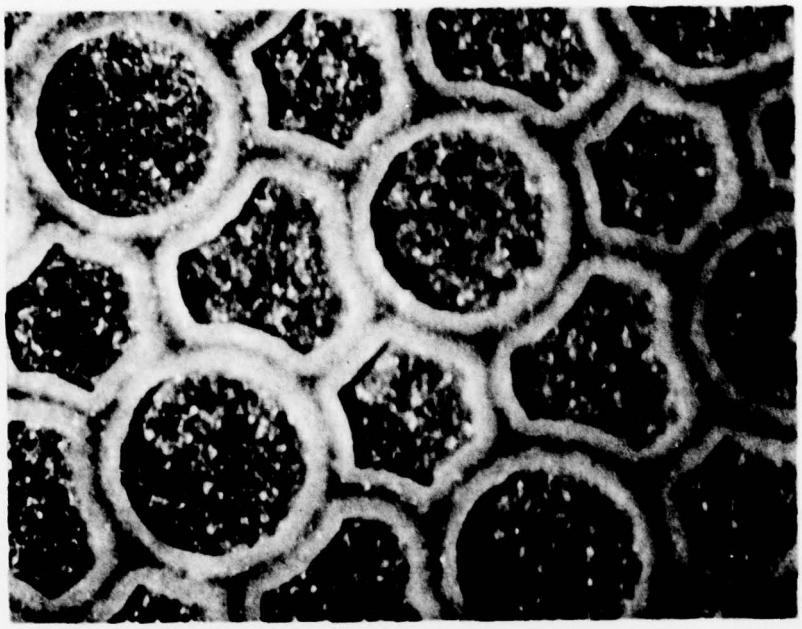

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Figure 7 Bonded grid assembly.

SECTION 3

CHEMICAL VAPOR DEPOSITION

Chemical vapor deposition is the primary process employed in producing the bonded grid. This technique is employed both to deposit the dense tungsten layer and the insulating layer of hafnium oxide. It was also used in an unsuccessful effort to deposit aluminum oxide. Ultramet; a local firm located at 12173 Montague Street, Pacoima, California 91331 and specializing in the chemical vapor deposition of refractory metals; agreed to attempt the experimental deposition of hafnium oxide and aluminum oxide as well. The early samples of hafnium oxide showed great promise, but the results have not always been repeatable. Preliminary efforts to improve this situation resulted in closer communications with Ultramet and the reservation of specific lots of raw materials for subsequent work. These measures gave some relief, but the need for further improvement was evident, and finally a carefully controlled experiment was initiated.

The deposition of a dense tungsten layer on the cathode surface did not present any particular problems. Ultramet routinely performs this activity in the course of manufacturing complex tungsten structures for other customers, and was able to produce excellent coatings. It was not possible to control the thickness of the coatings, as no means of monitoring the thickness during deposition was available; so a subsequent grinding operation was required. Figure 8 shows a cross section of a sample cathode coated with dense tungsten and ground to give the required coating thickness. The results are quite satisfactory.

The deposition of hafnium oxide presented much more of a problem. Ultramet's experience with the deposition of hafnium carbide[1] gave reason to believe that hafnium oxide could also be produced, but this required the extension of existing technology to new horizons. The very first efforts, even prior to launching this program, produced

1. R.B. Kaplan, "Chemical Vapor Deposition of Hafnium Carbide," Proceedings of the Third International Conference on Chemical Vapor Deposition (6/72).

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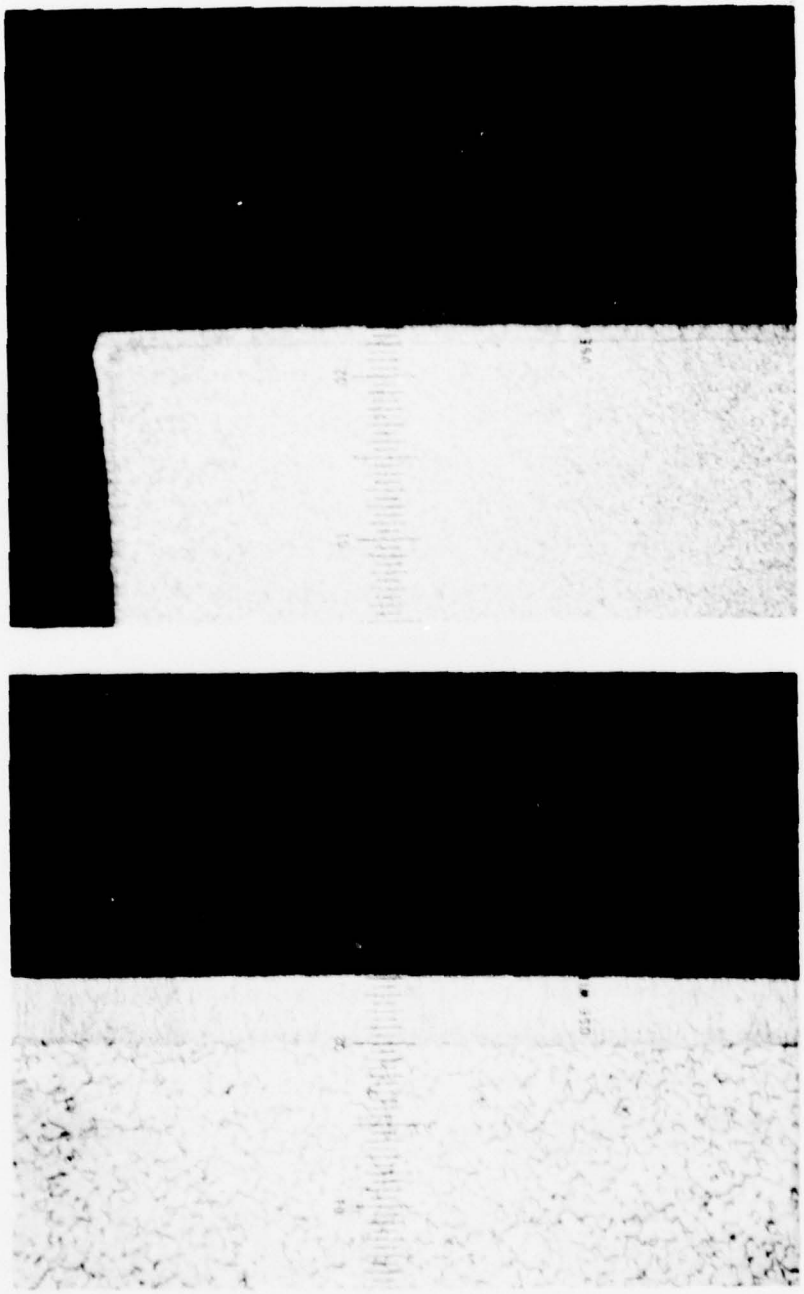

Figure 8 Dense tungsten coating obtained by chemical vapor deposition.

samples which appeared quite satisfactory, but subsequent attempts failed to duplicate the early success, at least not on a repeatable basis.

Figures 9 through 12 show several views of one of the first hafnium oxide samples obtained under this program. This part was intended for use in experiments to determine the dielectric properties of hafnium oxide, so a thin (2 micron thick) layer of molybdenum was sputtered over the insulator's surface to serve as a conductor. After initial testing an attempt was made to clear short circuits by applying a voltage across the insulator. This resulted in the creation of several damaged areas. Figure 9 shows an overview of the entire sample and Figures 10 and 11 show higher magnification pictures of the hafnium oxide coated surface. Figure 12 shows a metalographic section of the same part and reveals two major problems; the coating is too thin and it is not everywhere bonded to the tungsten substrate. Figure 13 shows a similar section from another sample which was obtained at the same time but which exhibited superior bonding.

These data were communicated to Ultramet and the coating process modified. Some later samples showed improved bonding, but an increased tendency towards columnar structure was also detected as can be seen in Figures 14 and 15. The significance of this structure was not determined, and subsequent results were again inconsistent. Several additional measures were then instituted to try and improve repeatability. Bottled air of known constant composition was substituted for ambient air, more careful records were maintained, and the remaining raw materials required were set aside for the program so that these factors would not vary. Although these steps did not produce a complete solution to the problems several useful samples were obtained.

Having inaugurated the above controls, several samples were prepared with the object of performing tensile strength tests to evaluate the

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Figure 9 Tungsten pellet coated with hafnium oxide.

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Figure 10 Hafnium oxide coating obtained by chemical vapor deposition.

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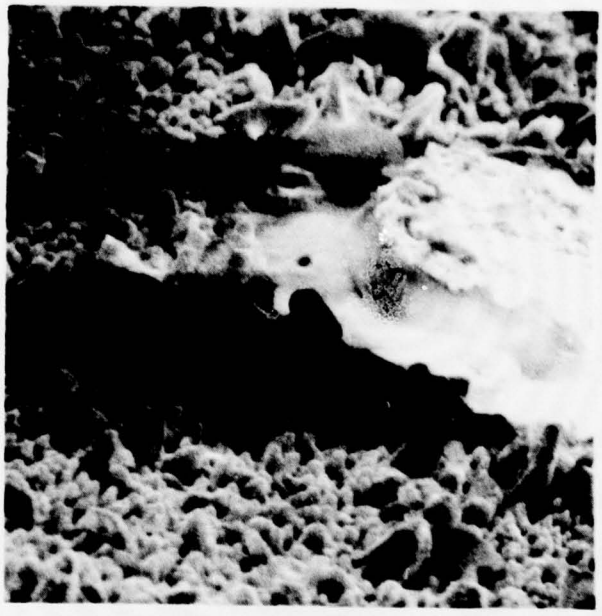

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Figure 11 Hafnium oxide coating obtained by chemical vapor deposition.

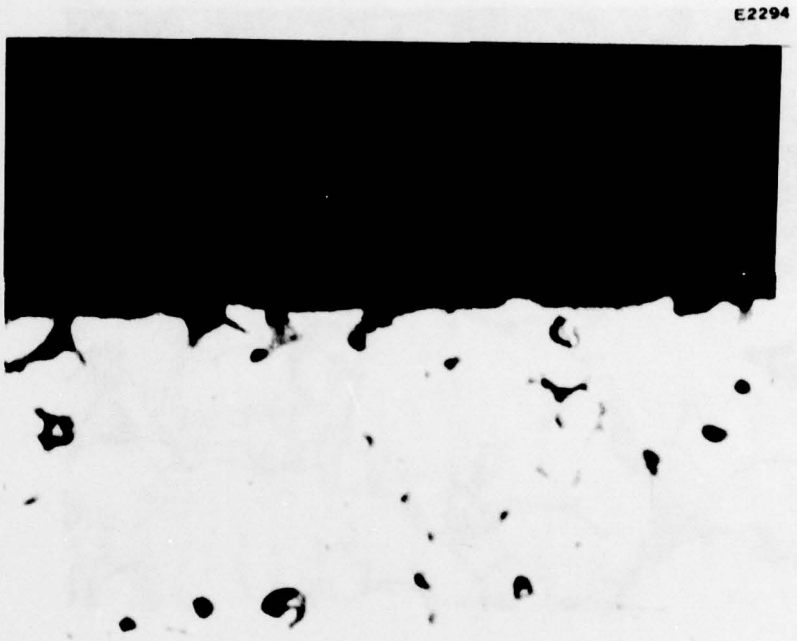

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Figure 12 Cross section of hafnium oxide coated tungsten pellet.

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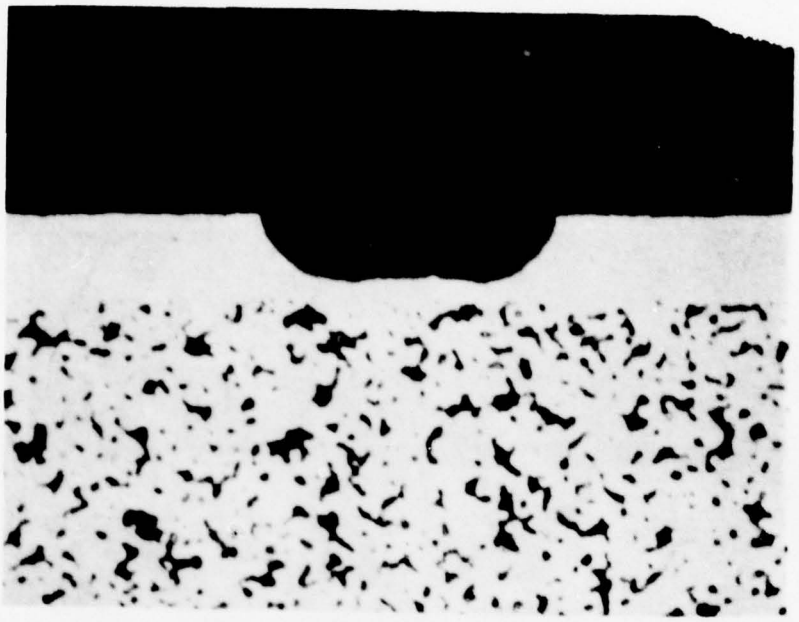
Figure 13 Cross section of hafnium oxide coated pellet.



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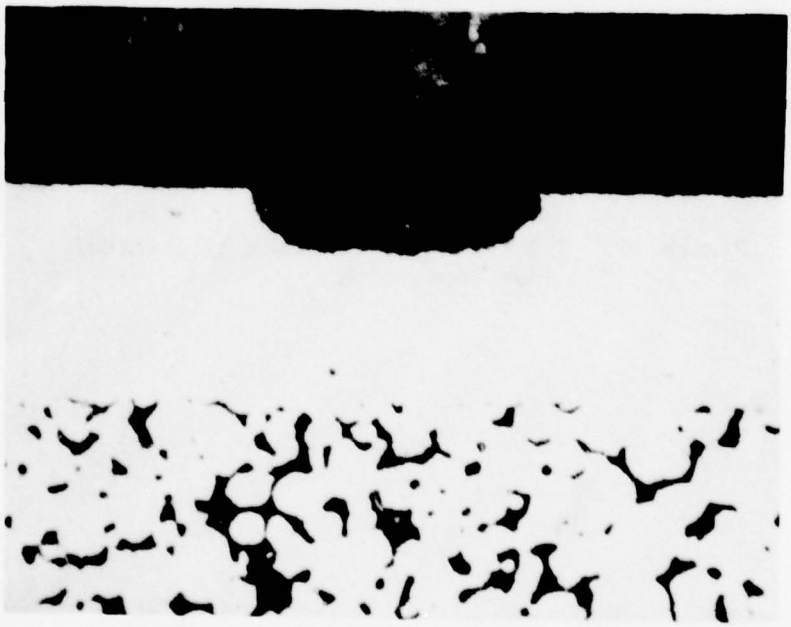

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Figure 14 Improved samples of hafnium oxide obtained by chemical vapor disposition.

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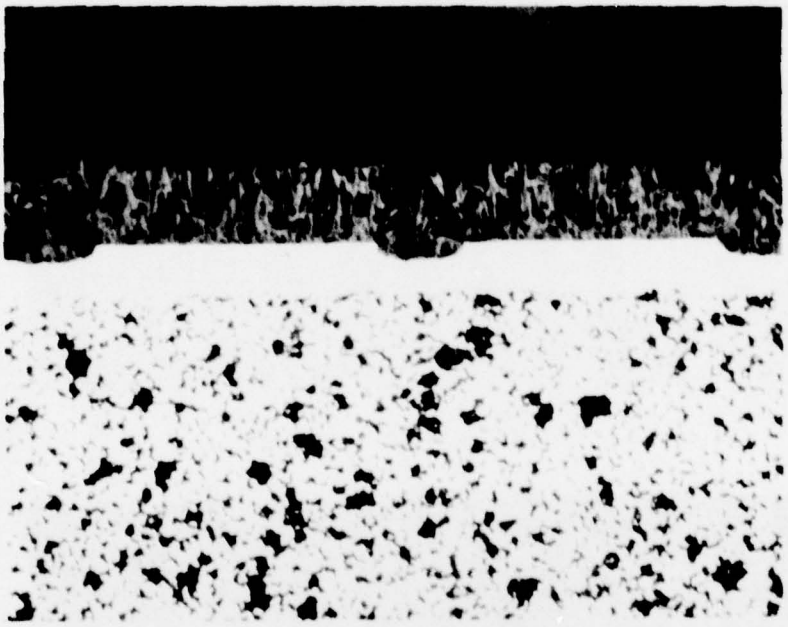

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Figure 15 Hafnium oxide obtained by chemical vapor deposition.

bond between hafnium oxide and tungsten. Eight samples were processed in two groups of four each with all parameters held constant, but the coatings differed between the two groups. A sample from one group is shown in Figure 16. The hafnium oxide failed to fill the groove which was to receive it, and the coating thickness was inadequate. A simple tape test administered to a sample from the same group suggested that the bond strength was insufficient. The second group, however, received a much better coating. A tape test failed to part the insulator from the tungsten substrate, and a calibrated tensile strength test showed the bond stength to be in excess of 2000 psi. A sample from this group, sectioned after further polishing and etching were accomplished, is shown in Figure 17. The insulator is seen to be in intimate contact with the cathode over a significant portion of their interface but, again, the rather coarse structure of the material is evident.

Further evaluation of the material obtained by chemical vapor deposition was severely complicated by the difficulty of controlling the process. The most reliable measurement of the insulator's resistivity indicated a value less than the published figure for bulk hafnium oxide by a factor of 10, but poor adhesion and the existence of defects in the insulator cast doubt upon this measurement. At this point in the development it was recognized that a comprehensive effort to control and optimize the deposition was essential, and an experiment consisting of a series of coating tests was initiated.

Although a large number of factors can influence the chemical vapor deposition of hafnium oxide the most significant parameters which can be conveniently varied are the specimen temperature, the system pressure and the hafnium flow rate. In addition, the condition of the surface on which the material is to be a deposited is of importance. In order to find the optimum combination of the three parameters fifteen trials were determined to be necessary based on a statistical analysis technique attributed to George G.E.P. Box of Wisconsin University.[2] Such

2. George E.P. Box and George C. Tiao, "Statistical Analysis and Design of Experiments", Final Report, contract AF-49 (638) - 1608 (10/63).

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Figure 16 Sample of hafnium oxide obtained by chemical vapor deposition showing failure to fill grooves.

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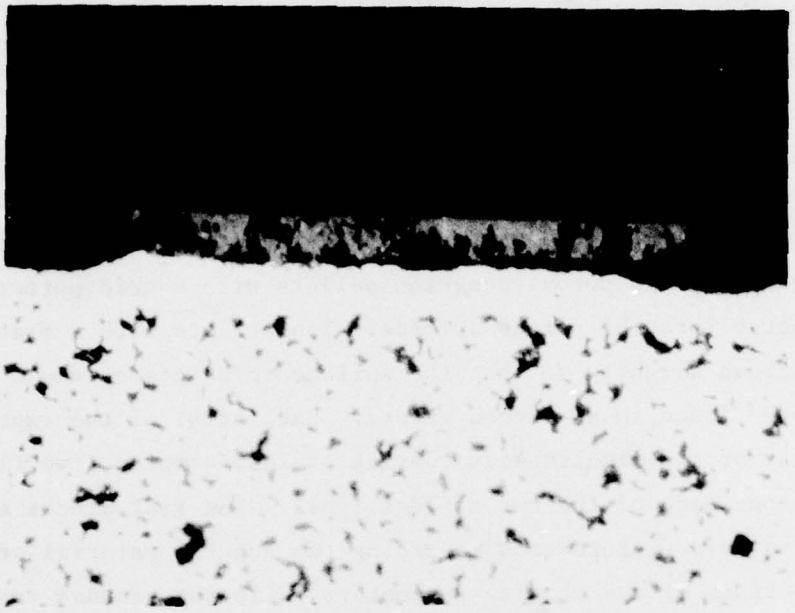

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Figure 17 Mechanically polished and plasma etched bonded grid assembly showing hafnium oxide obtained by chemical vapor deposition.

a series of experiments was planned and the parts ordered. Figure 18 shows the previous range of parameters reported by Ultramet and Figure 19 shows the schedule of coating parameters developed for the experiment. Special processing was instituted to insure the most favorable surface conditions, and during this processing potential problems were identified and resolved. Unfortunately time did not permit completion of the experiment.

The pieces to be coated in the experiment were of three types; porous tungsten pellets with a flat surface coating of dense tungsten, porous tungsten pellets with a grid pattern etched into the surface coating of dense tungsten, and porous tungsten pellets with a grid pattern etched directly into the porous surface. The pellets with a flat surface were etched slightly so that the surface to be coated would resemble that to be found in an etched groove. Each trial of the experiment is to consist of the simultaneous coating of four samples, two flat samples and one each of the two gridded types. The flat pieces are intended to provide information regarding the general material properties as functions of the coating parameters while the gridded samples are to allow evaluation of optimum samples under actual conditions. In preparing these pieces for the experiment, it was found that a contaminant was introduced during the mechanical polishing step, and that additional cleaning was required to eliminate it. Preparations for the coating experiment were delayed as a suitable cleaning process was developed. This cleaning is discussed more fully in the section of the report devoted to fabrication processes.

Attempts to produce layers of aluminum oxide bonded to the tungsten surface by the process of chemical vapor deposition met with little success. Apparently the difference between the coefficients of thermal expansion of the two materials prohibits the creation of layers with sufficient thickness to be of practical value in this application.

PARAMETER	PREVIOUS EXPERIENCE	RECOMMENDED RANGE
TEMPERATURE	1050–1175°C (BRIGHTNESS)	1000–1300° (BRIGHTNESS)
PRESSURE	24–74 MM OF Hg	24–120 MM OF Hg
HAFNIUM FLOW RATE	0.65–1.1 gm/MIN.	0.5–1.5 gm/MIN.

Figure 18 Chemical vapor deposition parameters. Values reported by Ultramet and recommended ranges for future studies.

TEST NO.	TEMPERATURE (°C)	PRESSURE (mm Hg)	Hf FLOW (gm/MIN.)
1	1273	111	1.41
2	1273	111	0.59
3	1273	32.5	1.41
4	1273	32.5	0.59
5	1027	111	1.41
6	1027	111	0.59
7	1027	32.5	1.41
8	1027	32.5	0.59
9	1300	72	1.0
10	1000	72	1.0
11	1150	120	1.0
12	1150	24	1.0
13	1150	72	1.5
14	1150	72	0.5
15	1150	72	1.0

Figure 19 Hafnium oxide chemical vapor deposition test conditions.

The possibility of grading the density of the deposited material in order to prevent spalling was considered but not pursued, efforts being directed towards the more promising work with hafnium oxide.

Chemical vapor deposition has been found capable of producing acceptable hafnium oxide coatings of usable thicknesses, but the process controls need to be better defined and regulated if reliable performance is to be expected. A series of experiments was initiated with this objective, and the work is continuing.

SECTION 4
FABRICATION TECHNIQUES

The fabrication of a bonded grid required the cultivation of many skills in addition to those required to perform chemical vapor deposition of materials. The basic approach outlined in Section 1 of this report and illustrated in Figure 1 involves precision machining and polishing steps, sputter deposition of copper, photomasking and chemical etching of copper, reactive plasma etching of tungsten, and sputter deposition of tungsten, all in addition to the chemical vapor deposition of both tungsten and hafnium oxide. A considerable effort was expended in adapting each of these processes to the present task, and several devices nearly satisfying the requirements were produced.

The first problem to be attacked was that of controlling the thickness of the deposited layers of tungsten and hafnium oxide. It was originally thought that chemical vapor deposition would produce coatings of predictable thickness; but this was found not to be the case, at least not with the available facilities and particularly if only a small quantity of parts were being processed. Since the dimension could not be controlled during deposition, it was resolved to apply excess material and then grind to the correct thickness. In order to assure success, two separate schemes were implemented. The first relied upon the use of a sacrificial fixture which was coated and ground along with the part. The fixture provided a reference from which the grinder could determine when the thickness was corrrect. The second method used parts manufactured to such close tolerances that the part itself could provide the needed reference.

Figure 20 shows the cross section of a piece coated and ground with the aid of the sacrificial fixture. The fixture featured a conical surface which was revealed as a circle of changing diameter as the grinding proceeded and which could be used as an indicator of grinding

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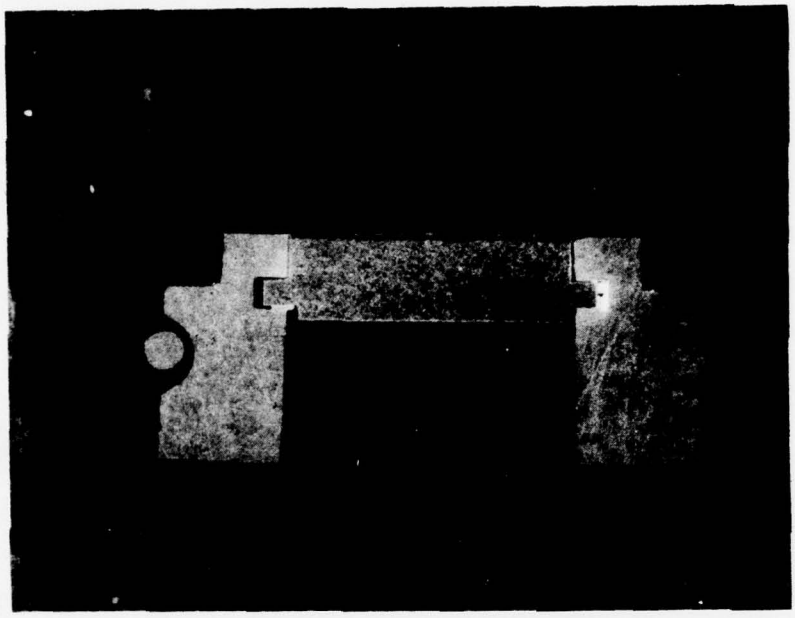

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Figure 20 Tungsten coated pellet ground in sacrificial fixture.

progress. The portion of that conical surface remaining after grinding is more readily apparent in the highly magnified view of Figure 21. A nonuniformity in thickness of the deposited layer is also apparent. This method was abandoned when the second technique produced superior results.

The second scheme is basically that outlined in Figure 1; the thickness of the cathode pellet was controlled within very tight tolerances, and then after each coating was applied, the excess material was removed by lapping while using the overall thickness of the part as a reference. Two variations of this approach were used. In one case dimensions were held within 0.00005" during each critical machining step while in the other they were held only to within 0.0002", but measurements were made to 0.00005" and subsequent design dimensions altered to compensate for any errors which were detected. The latter method severely complicated the flow of processing, but allowed greater flexibility when fabricating small quantities. In either case the thickness of the tungsten coating could be controlled within 0.0003" or better. The cross sectioned pellet displayed in Figure 8 was produced in this manner as were all subsequent pieces.

The above technique of course presumes that an excess of material has been deposited so that lapping will leave only this desired amount of material. This condition was met without difficulty in the case of tungsten deposition, but with hafnium oxide the coating thickness prior to lapping was frequently inadequate. Naturally the lapping process could not compensate for this deficiency, so frequently the hafnium oxide layers, and consequently the grid insulator, were thinner than desired.

A second presumption of the process, one which caused some difficulty, is that the back side of the cathode pellet remain free of material

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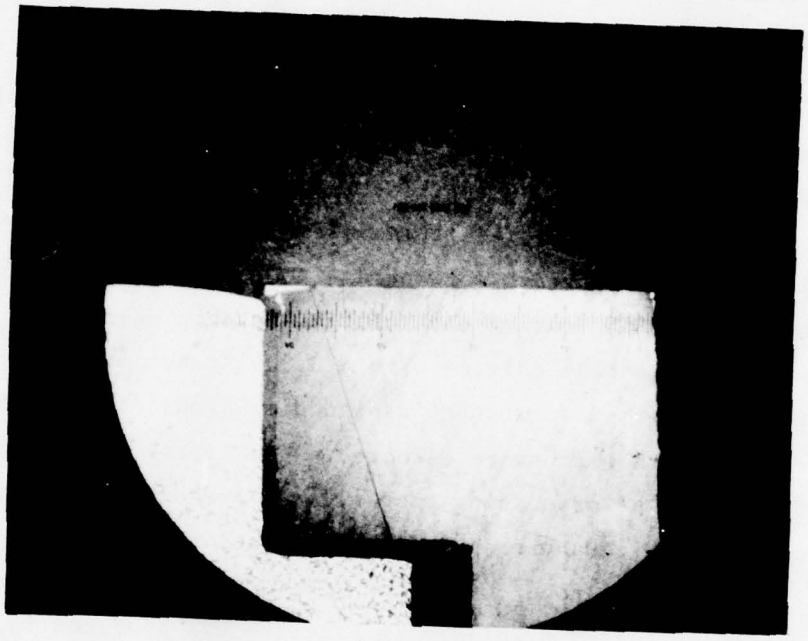


MAGNIFICATION = 50X

Figure 21 Tungsten coated pellet ground in sacrificial fixture.

during the deposition process. Any unwanted coating of the back side of the cathode contributes to the pellets height and can lead to an erroneous determination of the coating thickness. The use of a plated mask which could later be removed by selective chemical etching was considered to alleviate this problem. Assuming a suitable material could be found, this approach offered certain access to the datum surface for measurements; but it would have introduced a considerable amount of additional processing. In order to avoid this complexity and still achieve the desired result a more effective mechanical mask was sought. It was found that if the parts were placed upon a precisely machined flat surface during coating the unwanted deposits could be avoided.

Ultimately it is thought that the deposition process can be controlled to the degree necessary to yield coatings of precise thickness without further processing. Several possibilities have been proposed, but efforts for this program were directed along the avenue discussed above, since this seemed the most expedient means of achieving the required thickness control.

The next area of concern was the generation of an effective mask for the purpose of reactive plasma etching tungsten. This involved several steps, each requiring attention. Copper was selected as the material for the mask, and photolithographic techniques were used to produce the grid pattern in the mask.

The first step in producing the copper mask is to coat the tungsten surface with copper. A thickness of 5 microns was found to be adequate and was readily achieved by sputtering using conventional RF diode equipment employing substrate heating. The project was plagued by poor adherance of the masks, but a preliminary plasma etch of the surface in oxygen followed by sputter etching in argon was found to enhance adhesion.

With a layer of copper firmly bonded to the tungsten surface, the next step in producing the copper mask for reactive plasma etching is to photographically mask and chemically etch the copper. Standard solid state techniques were employed for this purpose throughout this program with satisfactory results, but the need for additional development was exposed.

Positive type KTI photo resist was applied to the copper coated pellets by applying drops of the resist while the part was spun on a table rotating at 4200 RPM. The part was located 1.0 inches from the axis of rotation as shown in Figure 22, and attached with double sided tape. After application of the resist the assembly was placed in a 90°C oven for 30 minutes to cure the resist.

A photo mask like that shown in Figure 23 was then used in conjunction with a Kasper Model 2001 wafer alignment machine to expose the resist. After exposure the resist was developed in KTI developer solution and rinsed in deionized water leaving the photo mask required for chemical etching of the copper. The copper was then etched in a bath of 40% nitric acid and rinsed in deionized water to remove the photo mask and give the desired copper mask for subsequent plasma etching of the tungsten. These processes are relatively standard and little work was done to optimize them beyond that required to achieve adequate adherance of the copper to the tungsten. It is thought that changes to these processes could yield more reliable results and greater resolution. In particular the use of ammonium persulfate to etch the copper might give some improvement.

Reactive plasma etching is required twice in the fabrication of a bonded grid assembly; in the first instance a copper mask is employed and dense tungsten previously applied by chemical vapor deposition is selectively removed to leave the grooves which are to receive the hafnium oxide insulator, and in the second instance the hafnium oxide serves as a mask as dense tungsten is removed to expose the porous tungsten

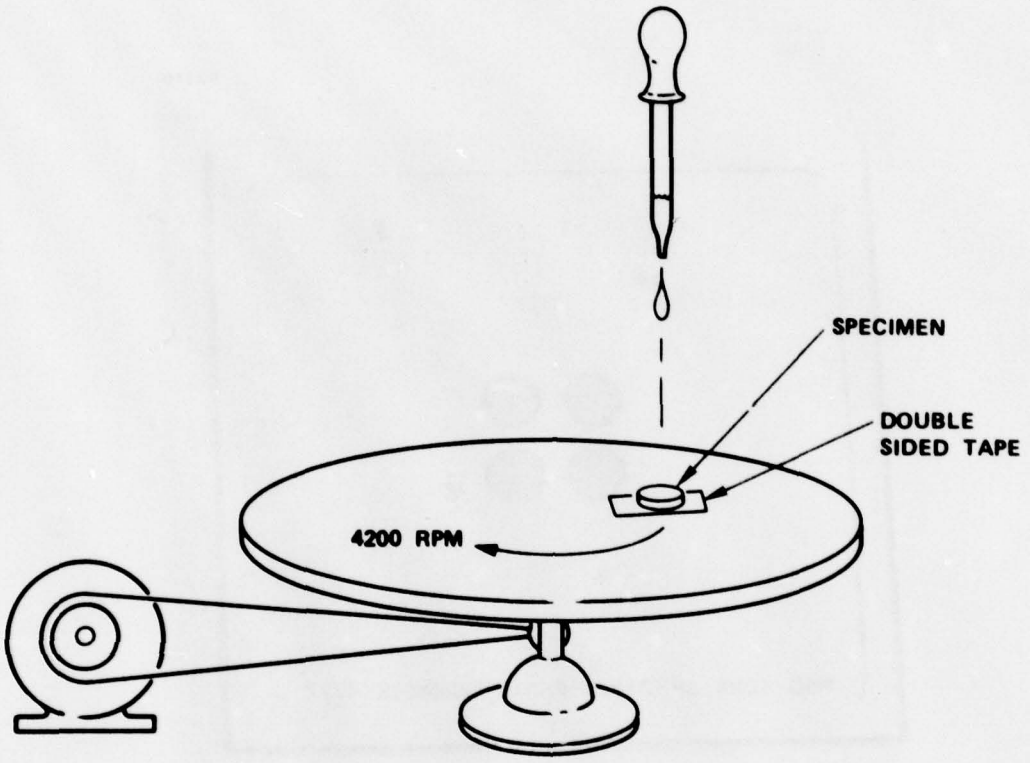


Figure 22 Rotating table for application of photo resist.

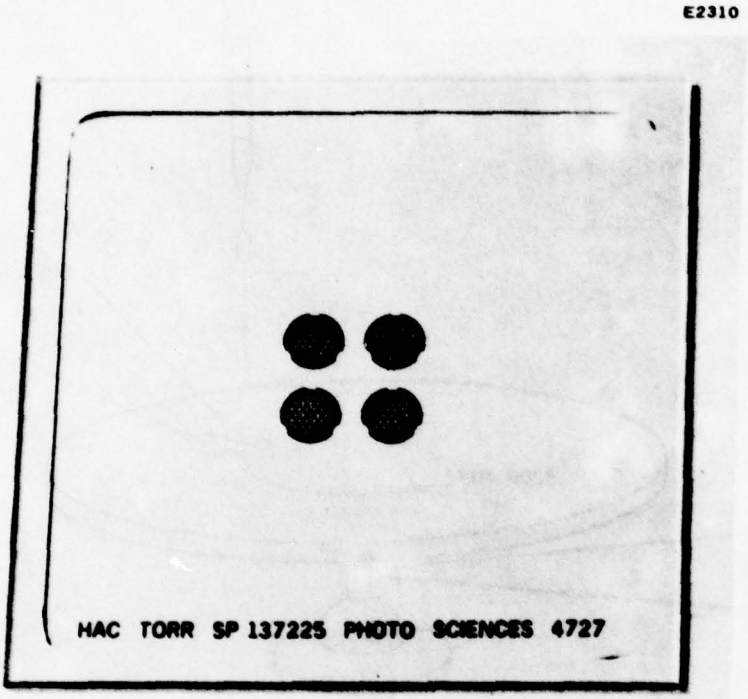


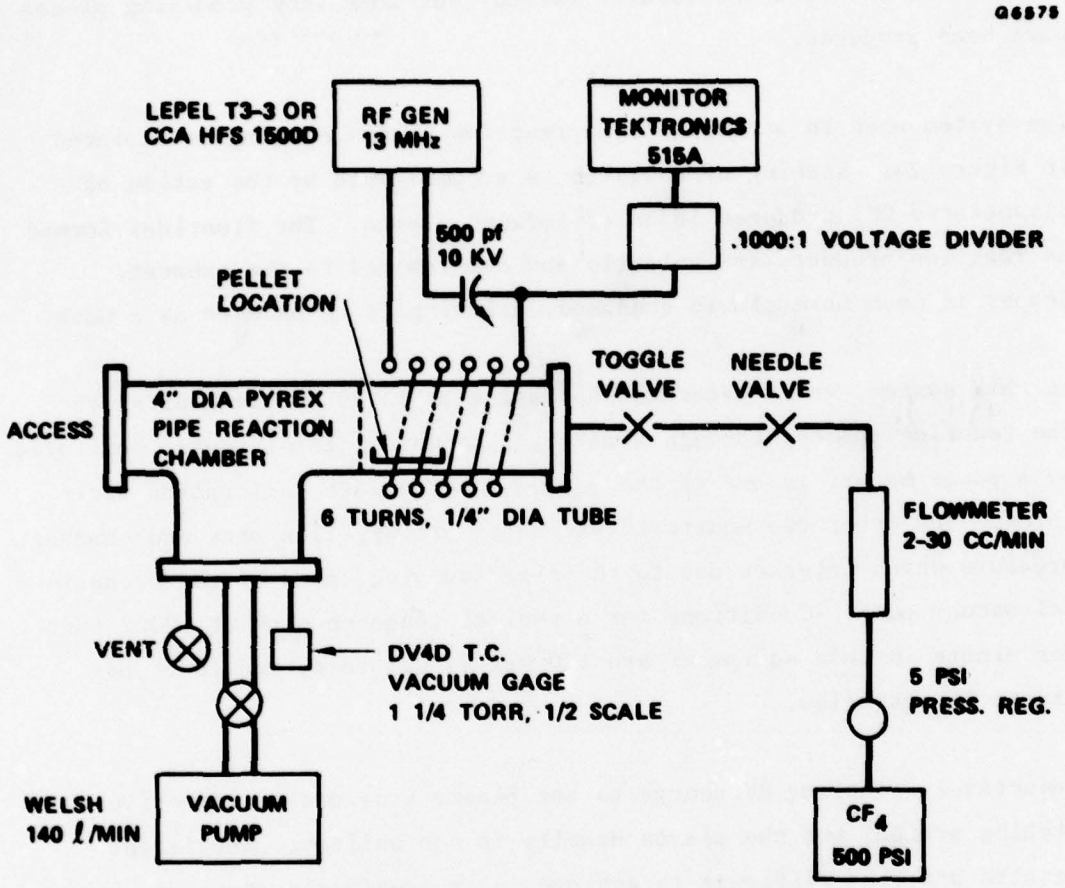
Figure 23 Bonded grid photo mask.

emitting surface. In the first case, with a copper mask, the results have been quite satisfactory so long as the mask is well bonded and the undercut accounted for. Experience with the hafnium oxide mask has been limited and the results varied, but some very promising pieces have been produced.

The system used to accomplish the reactive plasma etching is depicted in Figure 24. Etching of tungsten is accomplished by the action of dissociated CF_4 produced in an rf induced plasma. The fluorides formed as reaction products are volatile and are removed in the exhaust. Copper is much more slowly consumed, allowing it to be used as a mask.

In this system, an rf generator operating at 13.56 MHz is coupled to the reaction chamber through a matching network. Input power, monitored by a power meter, is one of the principal etch rate calibration variables. The other two monitored variables are CF_4 flow rate and chamber pressure which interact due to the fixed pumping speed of the mechanical vacuum pump. Conditions for a typical tungsten etch at .0002 inch per minute in this equipment are 120 watts, 0.5 torr, and 18 cc per minute CF_4 gas flow.

Inductively coupling RF energy to the plasma provides an effective etching action, but the plasma density is non-uniform. Consistent results are thus difficult to achieve, as a specimen's position within the reaction chamber influences the etch rate. Despite this handicap the process has proved to be an effective means of etching tungsten, and a well bonded copper mask has been found to provide adequate protection where material is not to be removed. Excellent results such as those displayed in Figures 25 and 26 were obtained when etching the grid pattern into the dense tungsten layer on the cathode surface. The photographs were made prior to removing the copper mask, and undercutting is clearly visible. Using a sputtered copper mask while etching to depths of .0020" or less in the dense tungsten obtained by chemical



TYPICAL OPERATING CONDITIONS

CF_4 FLOW	18 CC/MIN
PRESSURE	500×10^{-3} TORR
RF POWER	120 WATTS
IND. COIL VOLTAGE	1500 V PP

Figure 24 Reactive plasma etching apparatus.

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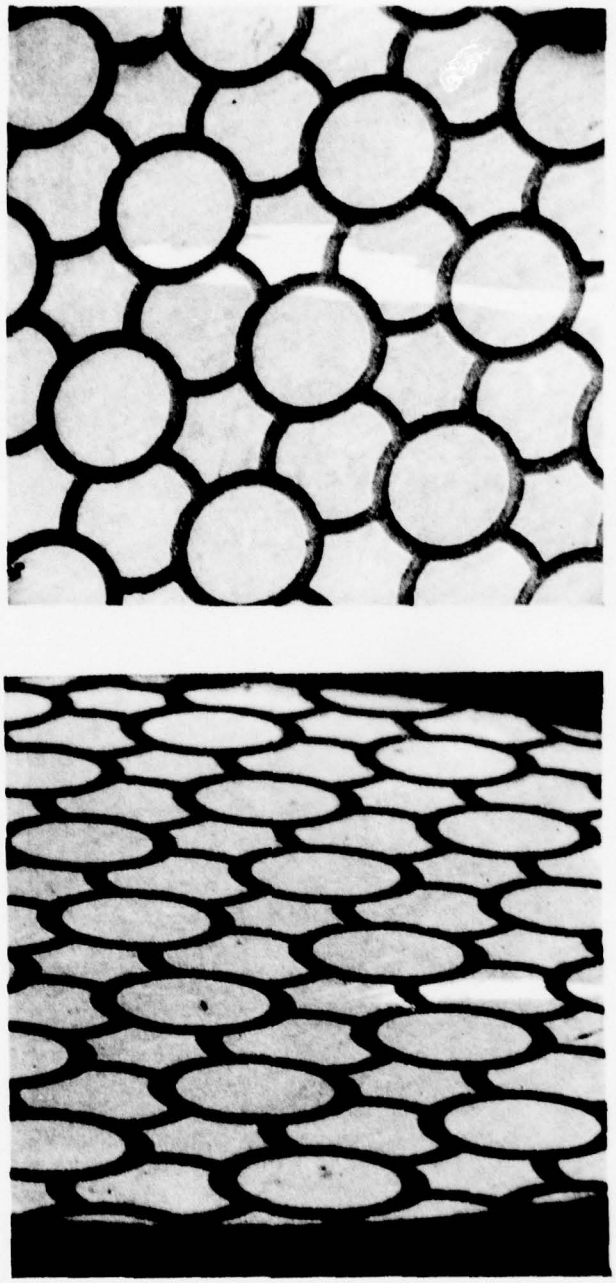


Figure 25 Bonded grid prior to removal of copper mask.

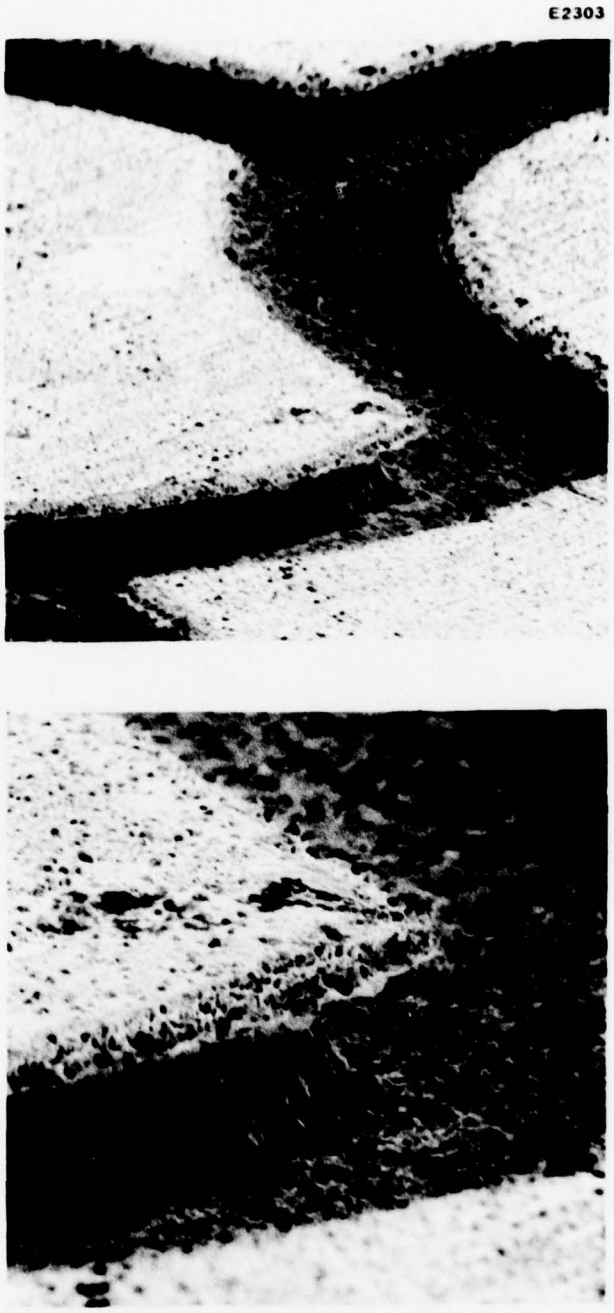


Figure 26 Bonded grid prior to removal of copper mask.

vapor deposition, the undercut was found to be approximately equal to the etch depth; and it was a simple matter to compensate by altering the mask.

In the final reactive plasma etching step which is intended to expose the surface of the porous tungsten cathodes, the hafnium oxide grid insulator serves as a mask while the dense tungsten is removed from between the elements of the grid. The procedure has been difficult to evaluate, since its success depends heavily upon the preliminary processes. Hafnium oxide was demonstrated to be relatively immune to attack by the reactive plasma, but some question remains concerning adhesion to the tungsten substrate. The grid shown in Figure 3 illustrates these facts. The final etch was conducted with frequent interruptions for inspection, and the process was terminated when separation of the insulator from the cathode surface was observed. In Figure 27 a non-adhering section of the grid insulator can be clearly seen. The groove behind the insulator indicates that the reactive plasma etch was terminated without removing enough material. Figure 4 shows the insulator in an area where the structure remained intact.

Several mechanisms were proposed to account for the difficulties in maintaining contact between the insulator and substrate during the reactive plasma etch. These included the possibilities of poor bonding achieved during the chemical vapor deposition of hafnium oxide and undercutting of the insulator or stressing of the bond during the etching process. Figure 28 shows a cross section of the assembly that was pictured in Figures 3, 4 and 28. It is evident that undercutting is not the problem, and that either the initial bonding was inadequate or the bond was subjected to excessive stress. It was concluded that improvement of the chemical vapor deposition technique should also favorably influence this critical etching process, and fabrication efforts were renewed with concern focussed on the deposition process.

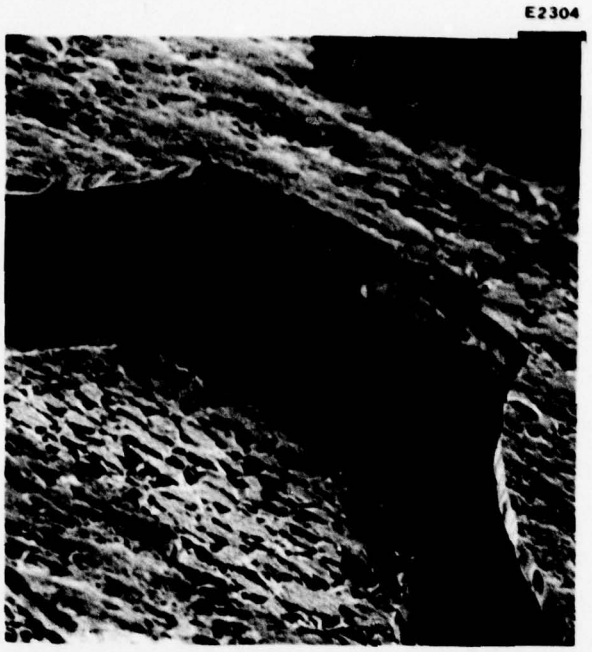


Figure 27 Non-adhering section of grid insulator.

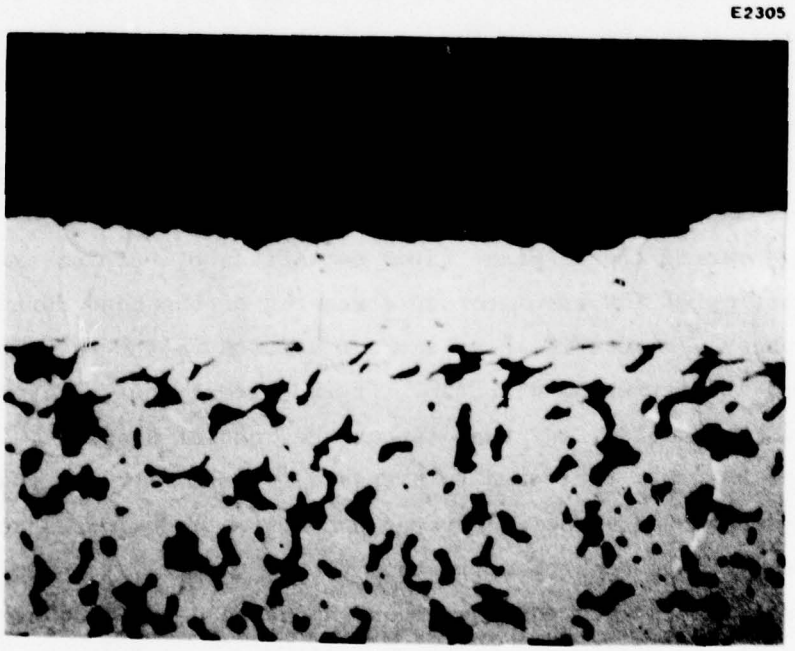


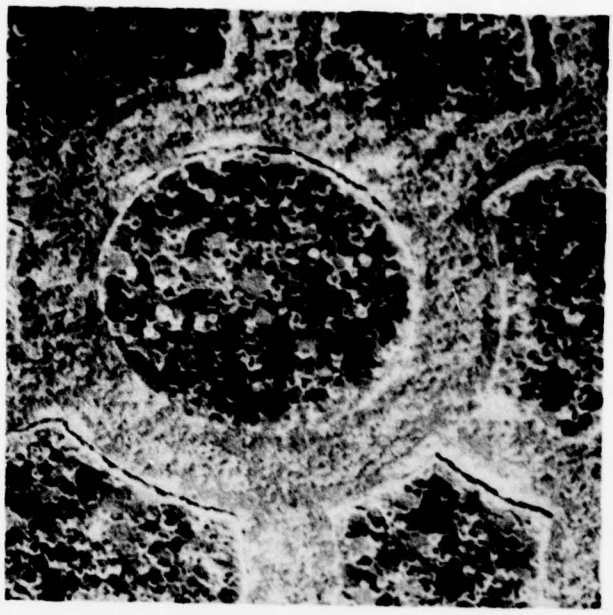
Figure 28 Cross-sectioned bonded grid assembly.

Figures 6 and 7 are photographs of the most recently completed assembly. Superior bonding of the insulator was achieved, so it was possible to completely remove the dense tungsten from between the grid elements and expose the porous tungsten cathode surface. The success achieved with this sample indicates that the reactive plasma etch technique is quite well suited to the task at hand, but that the material deposition techniques must be brought under better control.

It was necessary to examine the assembly with the scanning electron microscope in order to determine if the porous tungsten surface was actually exposed. Figures 29 and 30 show the cathode surface as the final reactive plasma etch progressed. In Figure 29 approximately 0.0014" of dense tungsten has been removed, but the cathode surface is not exposed. In Figure 30 all of the dense tungsten has been removed.

The deposition of a grid conductor on top of the insulator was accomplished by sputtering tungsten using conventional RF diode equipment employing substrate heating and presented no particular difficulties. Two concerns were the possibility of developing shorts between the cathode and the control grid and the possibility of the sputtered tungsten's inhibiting emission. Prior to its sectioning, a grid conductor was sputtered onto the device pictured in Figure 3 and resistance measurements made. The results are discussed more fully in the next section of this report, but as can be seen in Figure 37 the grid and cathode were not short circuited. Independent experiments demonstrate that several microns of tungsten could be sputtered onto the surface of a type B impregnated cathode without impairing emission.

It is intended that the fabrication steps discussed here be accomplished prior to the impregnation of the cathode. Several problems are foreseen in this regard, not the least of which is that the assembly must endure severe thermal stresses if conventional impregnation techniques are to be employed. While this may be feasible it represents an additional



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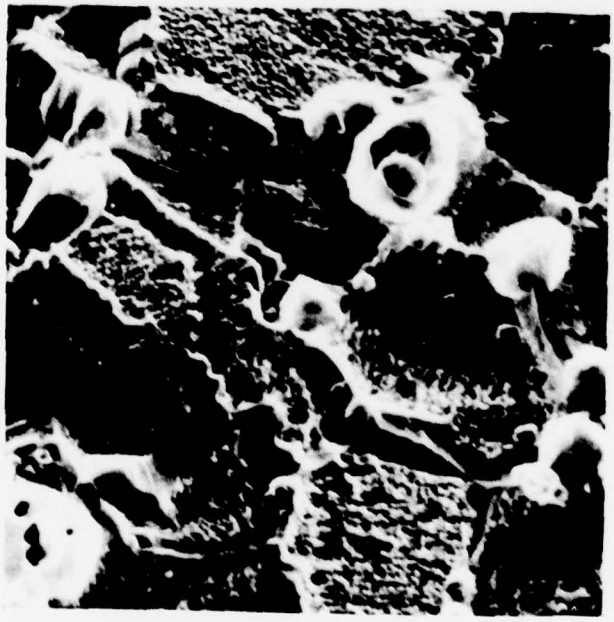

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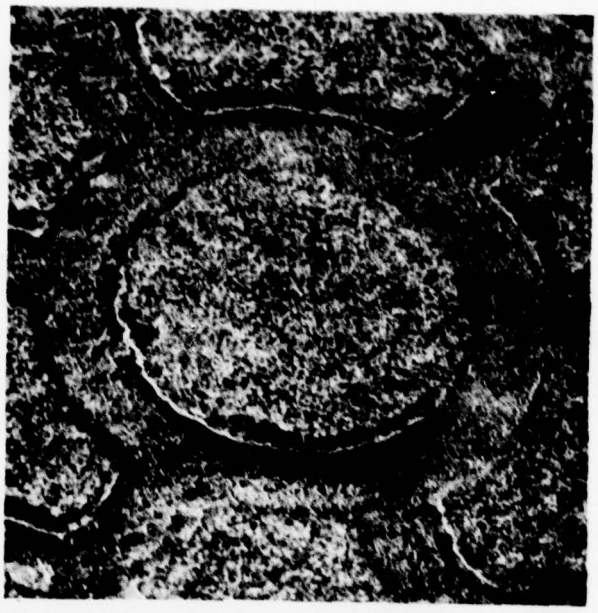
Figure 29 .Partially etched bonded grid assembly. Tungsten emitter not fully exposed.



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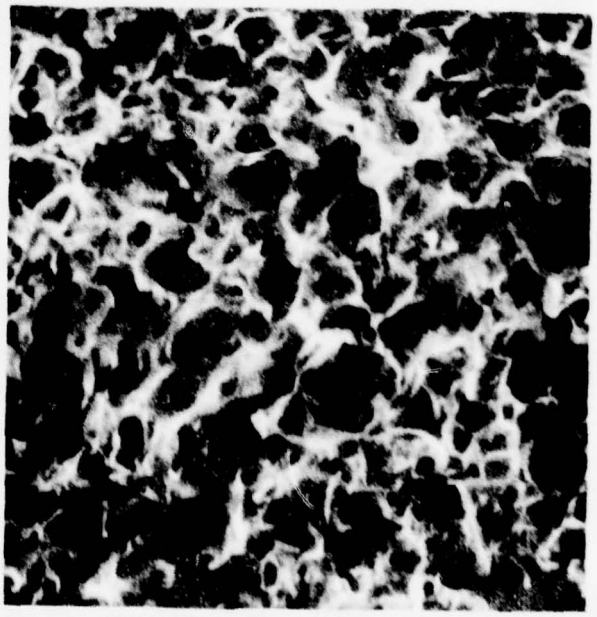

MAGNIFICATION = 1500X

Figure 30 Fully etched bonded grid assembly tungsten emitter fully exposed.

hazard, and it was decided to employ a different approach, at least temporarily. It was proposed that the bonded grid and cathode be fabricated and attached to a previously impregnated porous tungsten mount in a fashion similar to the "L" type cathode. This system was proved by assembling a non-impregnated porous tungsten pellet to an impregnated cathode and confirming emission. Satisfactory cathode activity was obtained, and the method deemed appropriate for application to bonded grid assemblies.

SECTION 5
MATERIAL EVALUATION

A control grid bonded to the surface of a cathode can be expected to operate at approximately the same temperature as the cathode, and thus the selection of a material to serve as the grid conductor is almost automatic, tungsten or molybdenum being natural choices. The control grid insulator, however, presents a much more difficult problem. The intent of this project was to capitalize upon the relatively stable high temperature properties of either hafnium oxide or aluminum oxide. A key concern was the evaluation of these materials to assess their electrical and mechanical characteristics as well as their degree of compatibility with conventional cathode technology.

Aluminum oxide has been used extensively in vacuum tubes of all descriptions and has been demonstrated to be suitable for use in conjunction with impregnated dispenser cathodes. Attempts to produce aluminum oxide by chemical vapor deposition were not successful, however, so a dielectric paste manufactured by the Transene Company of Rowley, was used instead. Coatings applied to cathodes prior to impregnation did not survive the impregnation processing, so tests were made by applying the material after impregnation. A cathode with a portion of its emitting surface coated by the dielectric paste was assembled into a diode and tested for emission. The cathode was quite slow to activate and failed after only a few days of operation. No further attempts were made to evalute aluminum oxide as a grid insulator.

Efforts to evaluate hafnium oxide were also hindered by the difficulty encountered in obtaining representative samples. Coatings were obtained by means of chemical vapor deposition, but the results were not at all consistent as is noted in Section 2 of this report. The problem was further compounded by the need to develop a technique of measurement. The best results indicated that the material's resistivity is

1500 Ω-cm at a temperature of 1050°C which is a factor of 10 less than that reported for bulk hafnium oxide.[3]

Measurement of the material's relative dielectric constant was not accomplished at temperatures in access of 400°C, but was found to be between 10 and 20 for temperatures less than 400°C.

In attempting to measure the resistance and capacitance it was found to be imperative that contact to the insulator be made by means of a conductive layer in intimate contact with the insulator's surface. The bonding of the insulator to the substrate provided one such interface, and it was found that a 2 micron thick sputtered layer of tungsten or molybdenum could be used to contact the opposite face. Measurements made using this technique were found to be much more reliable and repeatable than were those made with contact established only by pressure contact.

Initially simple flat pellets were to be coated with hafnium oxide in order to provide a sample uncomplicated by considerations of geometry so that resistance and capacitance might be readily evaluated. Such samples were prepared and tested, but the erratic quality of the coatings, particularly of the early coatings, precluded the acquisition of much meaningful data. Efforts to evaluate these first samples did, however, reveal particular difficulties with the measurement and resulted in the development of workable measuring techniques.

It became clear that a series of meter readings could not provide adequate information about the material's resistivity. The resistance of the device exhibited both a temperature and voltage dependence which necessitated the instantaneous collection of data if any understanding was to be achieved. The first device was, in fact, damaged due to an inability to quickly evaluate its condition. The circuit of Figure 31

3. U.S. Touloukian, editor, Thermophysical Properties of High Temperature Solid Materials, Vol 4, Part 1 (Macmillan Company, N.Y. 1967).

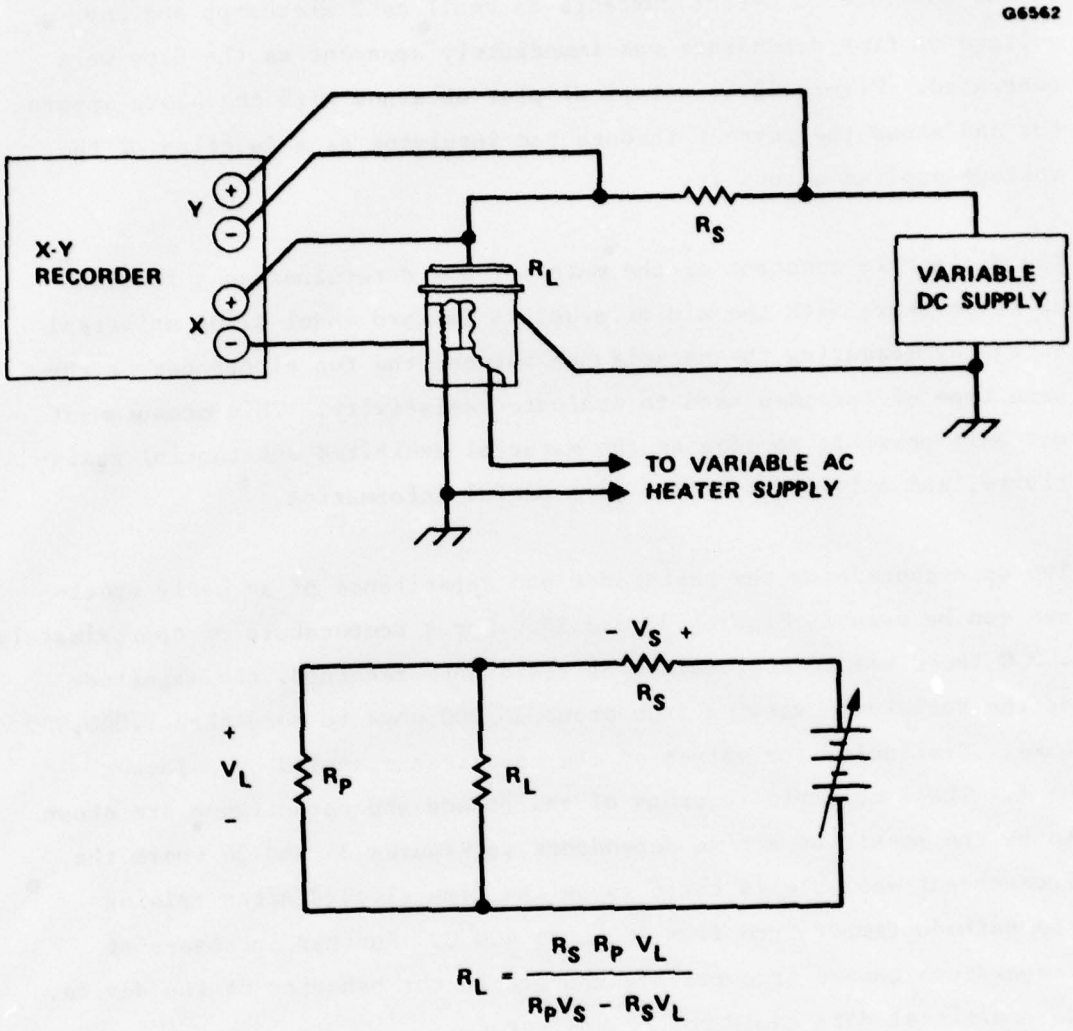

Figure 31 Circuit used to measure current through the grid insulator and to determine grid insulator resistance (R_L).

was adopted for subsequent resistance measurements. With this technique it was possible to detect currents as small as 2 microamps and any voltage or time dependence was immediately apparent as the data were generated. Figure 32 is a typical plot obtained with the above apparatus and shows the current through the insulator as a function of the voltage applied across it.

The dielectric constant of the material was determined as a function of temperature with the aid of a Helett Packard model 4260A universal bridge by measuring the capacitance between the two electrodes of the same type of specimen used to evaluate resistivity. This measurement was only possible so long as the material exhibited substantial resistivity, and only one specimen gave useful information.

The data concerning the resistance and capacitance of an early specimen can be seen in Figures 33 and 34. For a temperature of approximately 400^{o}C there are several values of resistance recorded, the magnitude of the resistance varying from about 10,000 ohms to more than 1,000,000 ohms. Similarly, the values of the capacitance varied by a factor of 4. These multiple readings of resistance and capacitance are shown to be the result of a time dependence in Figures 35 and 36 where the independent variable is taken to be the time elapsed after raising the cathode temperature from 300^{o}C to 400^{o}C. Further increases of temperature caused irreversible changes in the behavior of the device, so additional data could not be collected.

The relative dielectric constant of the hafnium oxide at room temperature was calculated from the data in Figure 34 to be 11. The calculation was based upon a parallel plate model, and considerable margin for error was introduced by uncertainties as to the coating thickness and the degree of bonding achieved between the hafnium oxide layer and the tungsten substrate. As the temperature increased the capacitance also increased, indicating an increase in the dielectric constant.

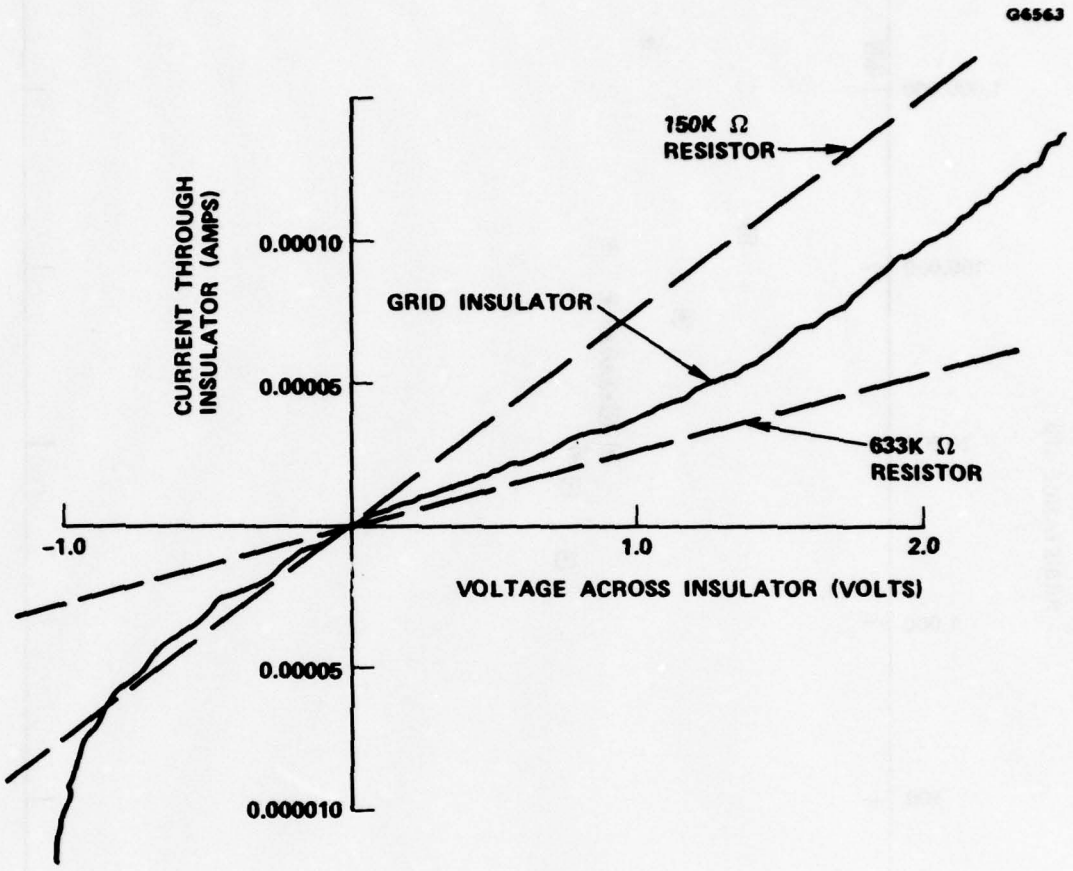

Figure 32 Typical plot of insulator current vs. voltage.

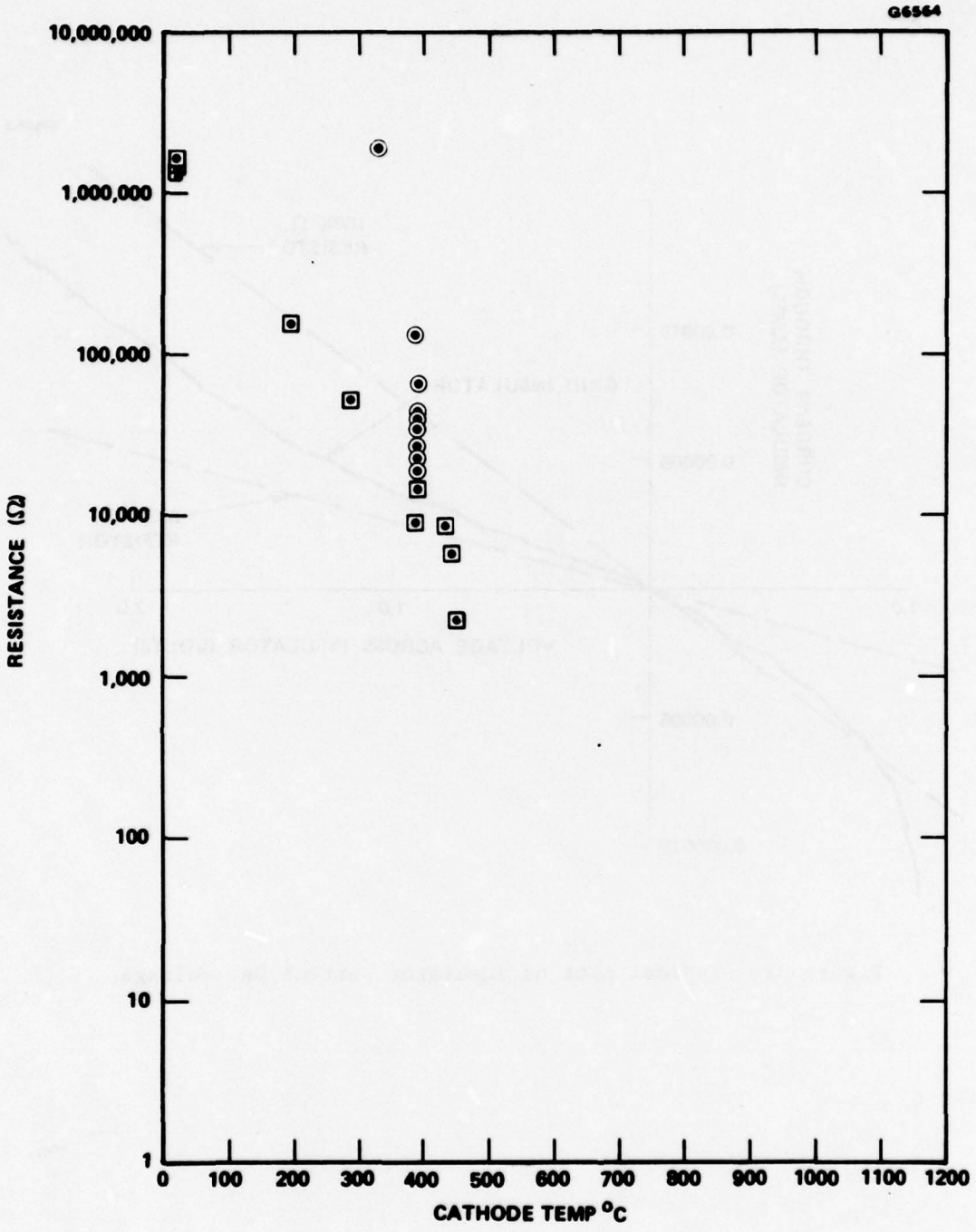

Figure 33 Hafnium oxide resistance bonded grid test device #4.

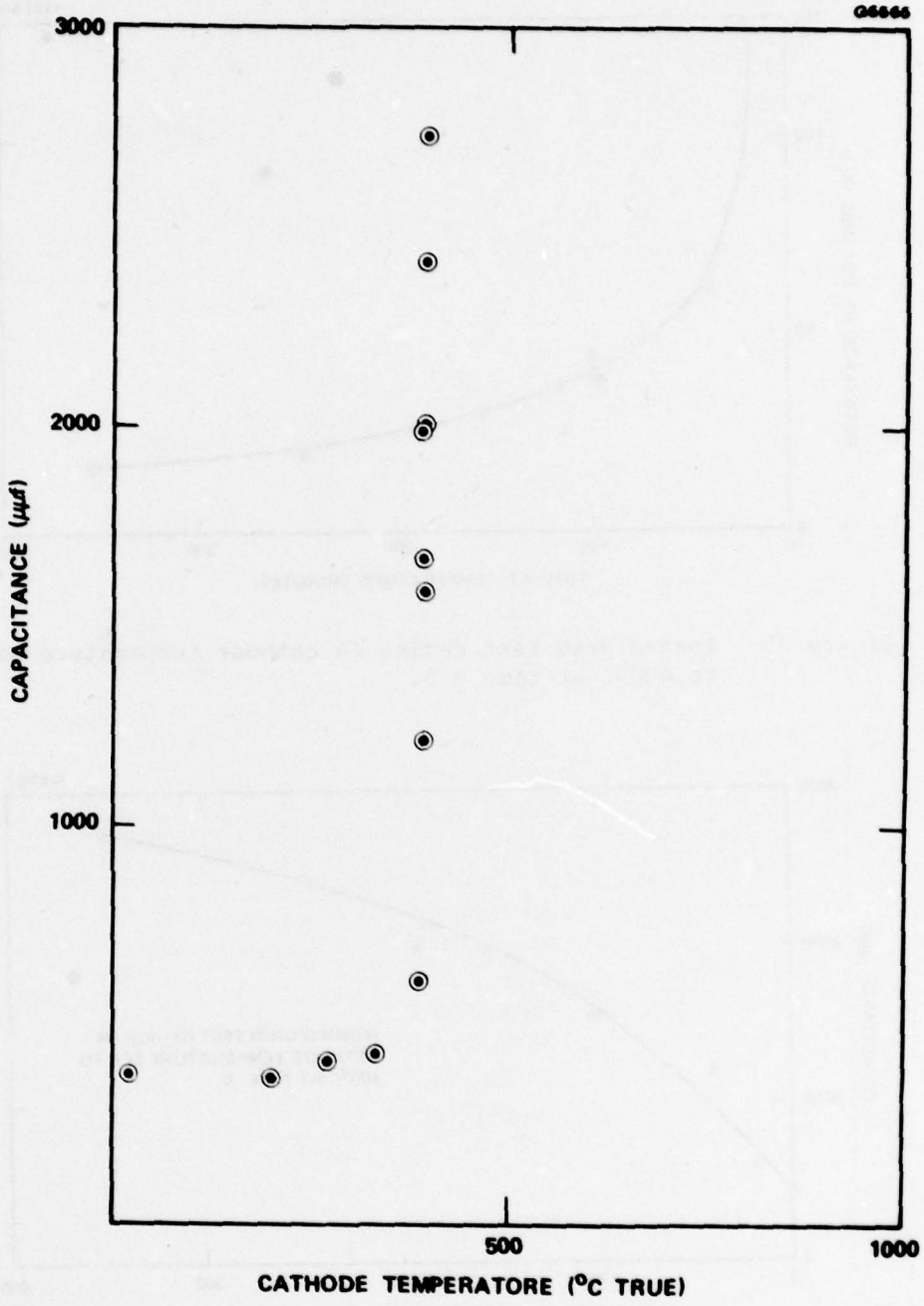

Figure 34 Hafnium oxide permitivity bonded grid test device #4.

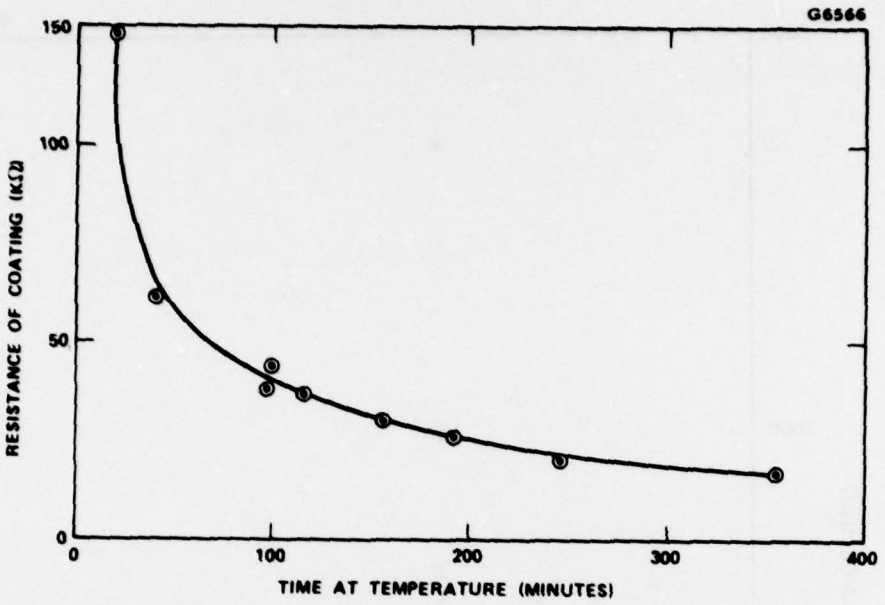

Figure 35 Bonded grid test device #4 cathode temperature set to 400°C at time = 0.

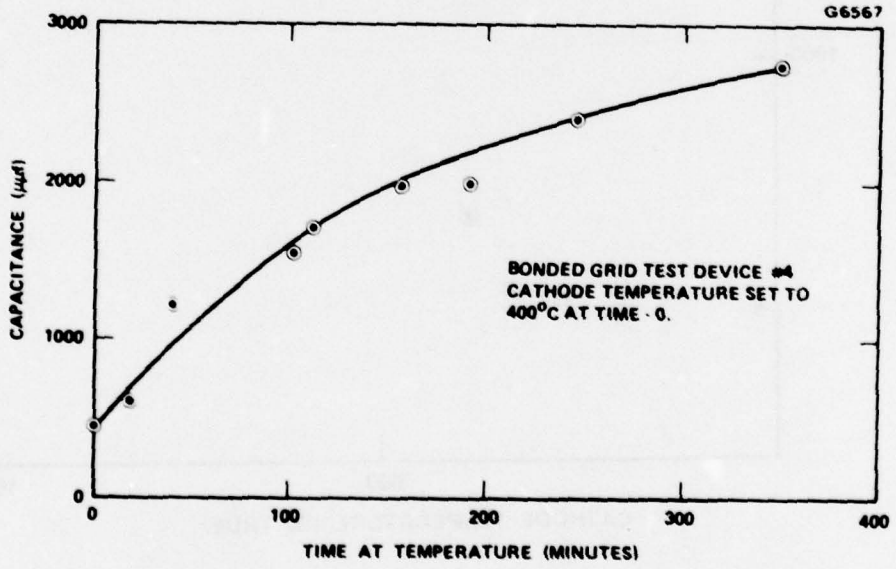

Figure 36 Bonded grid test device #4 cathode temperature set to 400°C at time = 0.

When the temperature of the specimen was increased to 400°C the relative dielectric constant immediately rose to 17 and continued to increase with the passage of time. Although the results of these measurements were not conclusive the value measured was approximately as expected, and is such as to result in reasonable grid to cathode capacitances. A more reliable measure of capacitance is contingent upon producing more satisfactory samples.

The anomalous characteristics exhibited by the samples of hafnium oxide prepared on flat tungsten pellets are thought to represent defects in the particular samples tested rather than an inherent deficiency of hafnium oxide. Upon sectioning, the hafnium oxide layer was found to be only 0.0002" thick and the adherence of the coating questionable, separation being evident in some areas. It was concluded that the performance irregularities observed were most probably due to localized defects in the coating, and efforts continued towards fabrication of a gridded device.

The first gridded assembly produced, that shown in Figure 3, was not capable of emission but provided an additional vehicle for measuring the resistance of hafnium oxide. Figure 38 displays the resistance between its cathode and grid as a function of temperature. The resistivity at an operating temperature of 1000°C was found to be 2750 Ω-cm, falling to 1500 Ω-cm at a temperature of 1050°C. This value is again less than the expected value for bulk material by a factor of 10, but still provides a degree of isolation between grid and cathode. Improvements in the chemical vapor deposition techniques are expected to increase the resistance obtainable by elimination of flawed areas within the coating.

The question of compatibility with cathodes is two fold. As proposed the insulator must not only be compatible with the operation of the

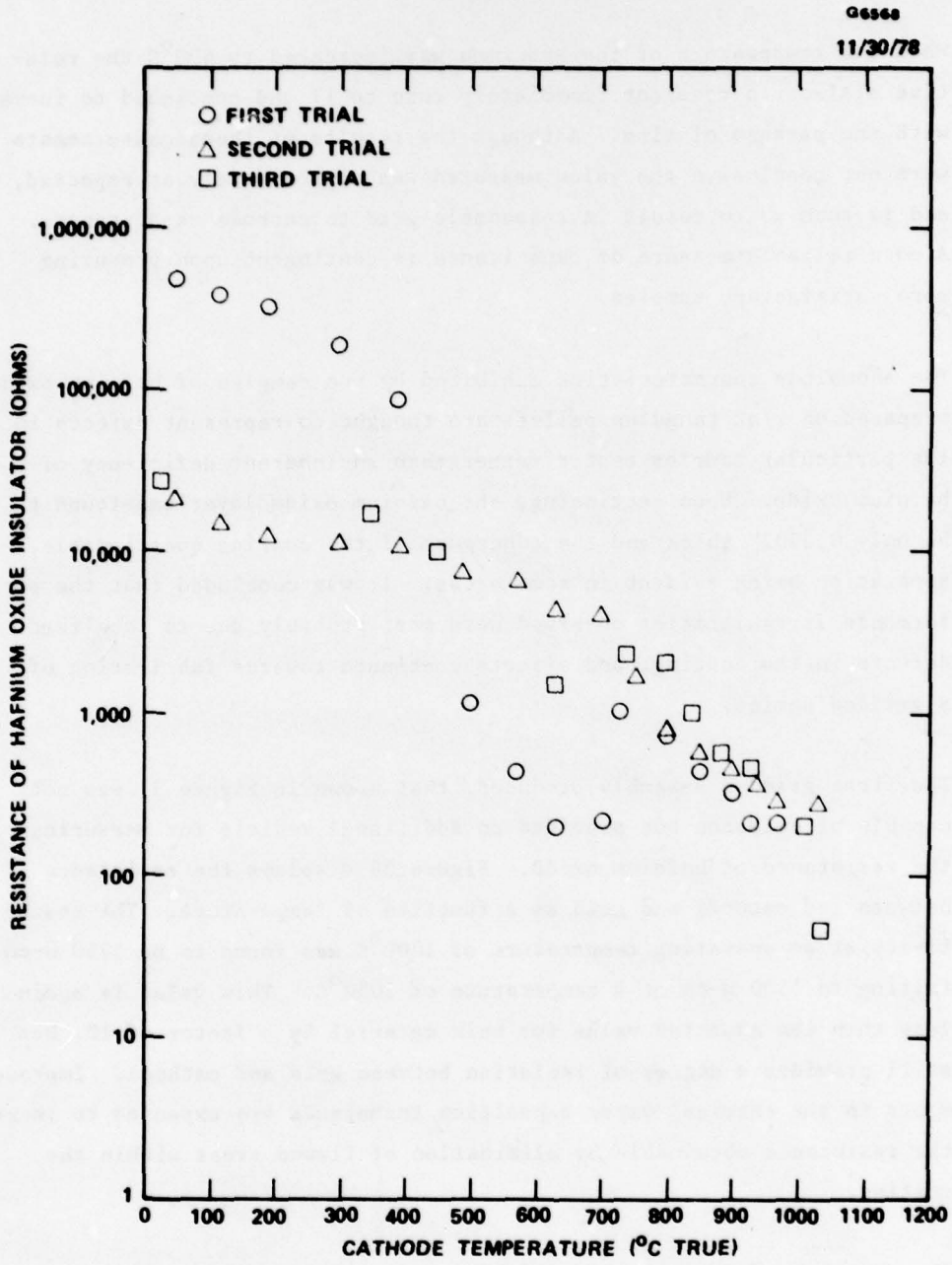

Figure 37 Grid insulator resistance.

cathode, it must be able to withstand the cathode impregnation temperatures as well. Samples of hafnium oxide deposited on a tungsten pellet were subjected to temperature excursions like those required for impregnation, and a bonded grid assembly was assembled into a triode and emission testing initiated.

Figures 38 and 39 are photographs of a hafnium oxide sample before and after being subjected to the impregnation temperature. After coating the part was placed in a fast cycle furnace and its temperature raised to 1650°C (Brightness) for 30 seconds and then quickly cooled to the ambient temperature. A tape test administered after the temperature cycling indicated that the bond to the cathode was still adequate although the photographs reveal an alteration in the structure of the material. These observations weighed heavily in the decision to employ an "L" type cathode configuration in initial activation experiments.

The bonded grid assembly shown in Figures 6, 7, 29, and 30 was selected for use in tests to establish the compatibility of the grid structure with cathode emission. The device was laser welded to a previously impregnated cathode and assembled into the parallel plate device depicted in Figure 40 which contained a special ring to contact the grid. The entire assembly was then placed in a vacuum chamber and baked out at approximately 100°C until the pressure was reduced to 1×10^{-7} torr. The chamber was then cooled and the cathode temperature slowly elevated, again keeping the pressure at or below 1×10^{-7} torr. After 192 hours of operation the cathode temperature reached 1000°C (brightness on tungsten) and was maintained at this level for activation and initial testing.

After 48 hours at 1000°C (brightness) the cathode began to emit, the current being only a few microamps, and the temperature was increased to 1040°C . With time more impregnant migrated to the surface of the bonded grid assembly, and the emission increased.

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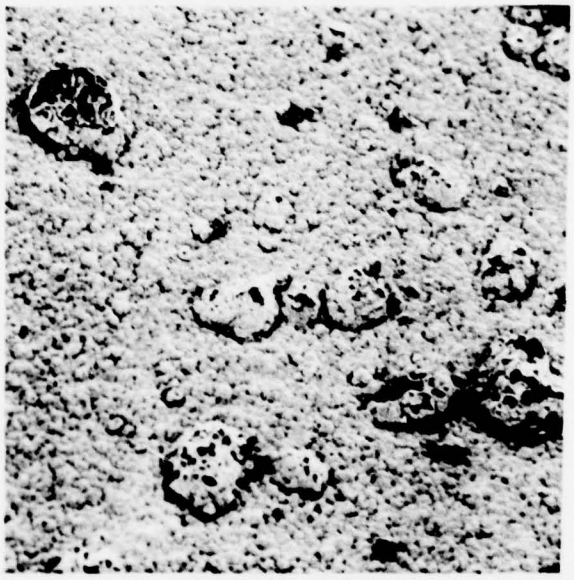

MAGNIFICATION = 300X

MAGNIFICATION = 15X

Figure 38 Hafnium oxide clad tungsten pellet prior to exposure to impregnation temperatures.

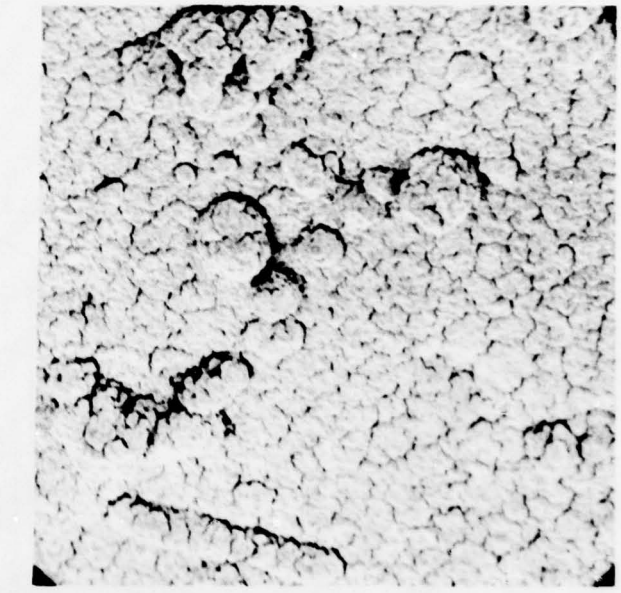

Figure 39 Hafnium oxide clad tungsten pellet after exposure to impregnation temperatures.

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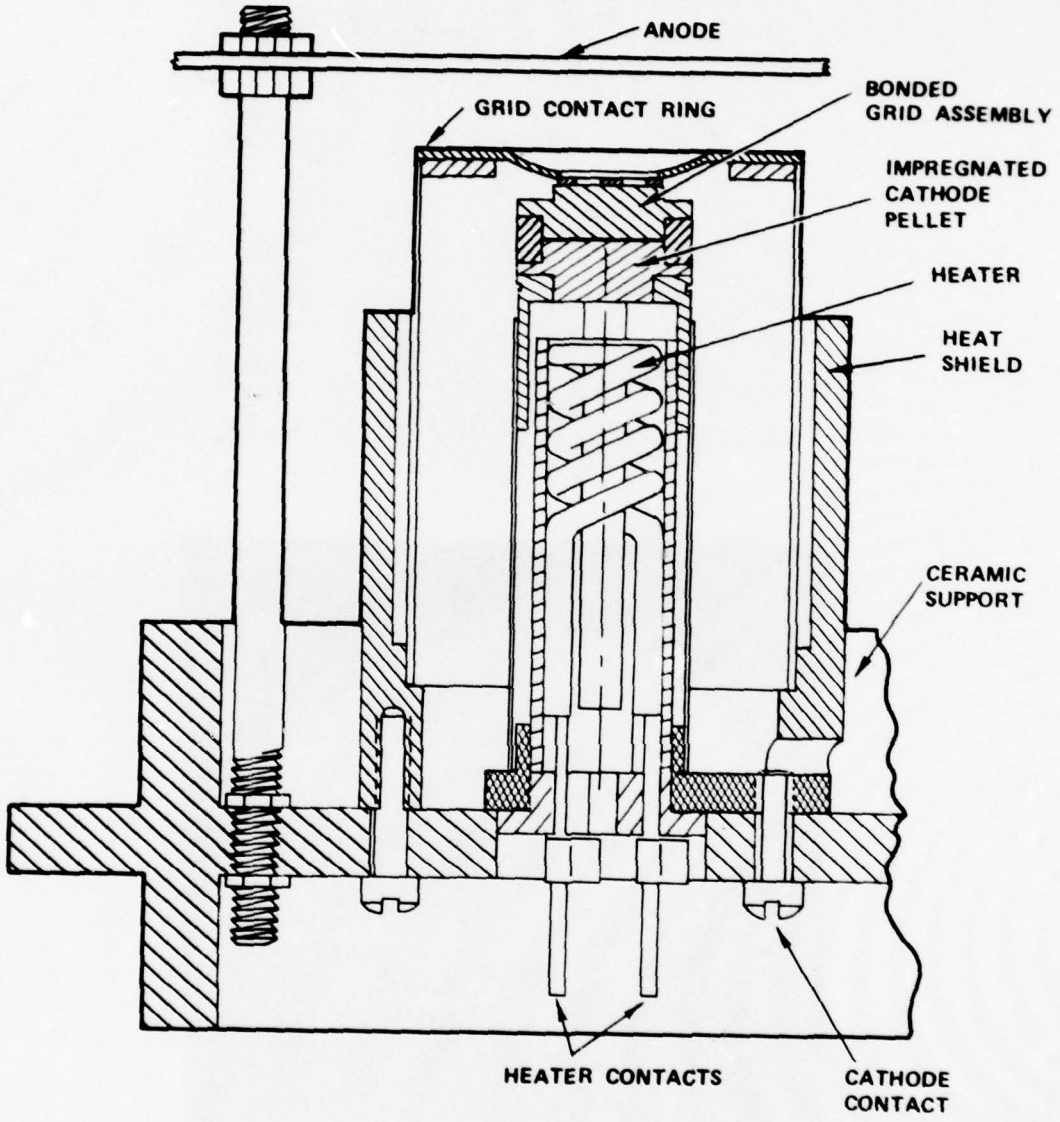

Figure 40 Bonded grid triode assembly.

The device was able to sustain .138 Amp/cm^2 after 168 hours at the operating temperature. Figure 41 is a plot of the current collected from the cathode as a function of the voltage applied between anode and cathode. The control grid voltage was maintained as a constant fraction of the anode to cathode voltage, so space charge limited emission should be characterized by a linear relationship between current raised to the two thirds power and voltage. The curve is seen to satisfy this requirement so long as the current emitted did not exceed 17 milliamps. This would correspond to a current density of .138 A/cm^2 assuming that all of the exposed cathode surface contributed to emission. It remains to be seen if the cathode will continue to activate if maintained at its present temperature or if, barring that, an increase in temperature will significantly improve cathode performance. The fact that emission has been obtained in the presence of hafnium oxide is encouraging, but additional work is required to demonstrate conclusively that it is compatible with cathode performance.

Of the two materials considered for this project only one, hafnium oxide, proved to be obtainable in a useable form. The evaluation of this material was complicated by the uncertainty of the deposition technique, but its potential for this application was demonstrated. The material obtained by chemical vapor deposition was found to exhibit substantial resistivity even when elevated to typical cathode operating temperatures and is thus a suitable choice as grid insulator. Furthermore the preliminary results of emission tests suggest that the material will prove to be compatible with normal cathode performance.

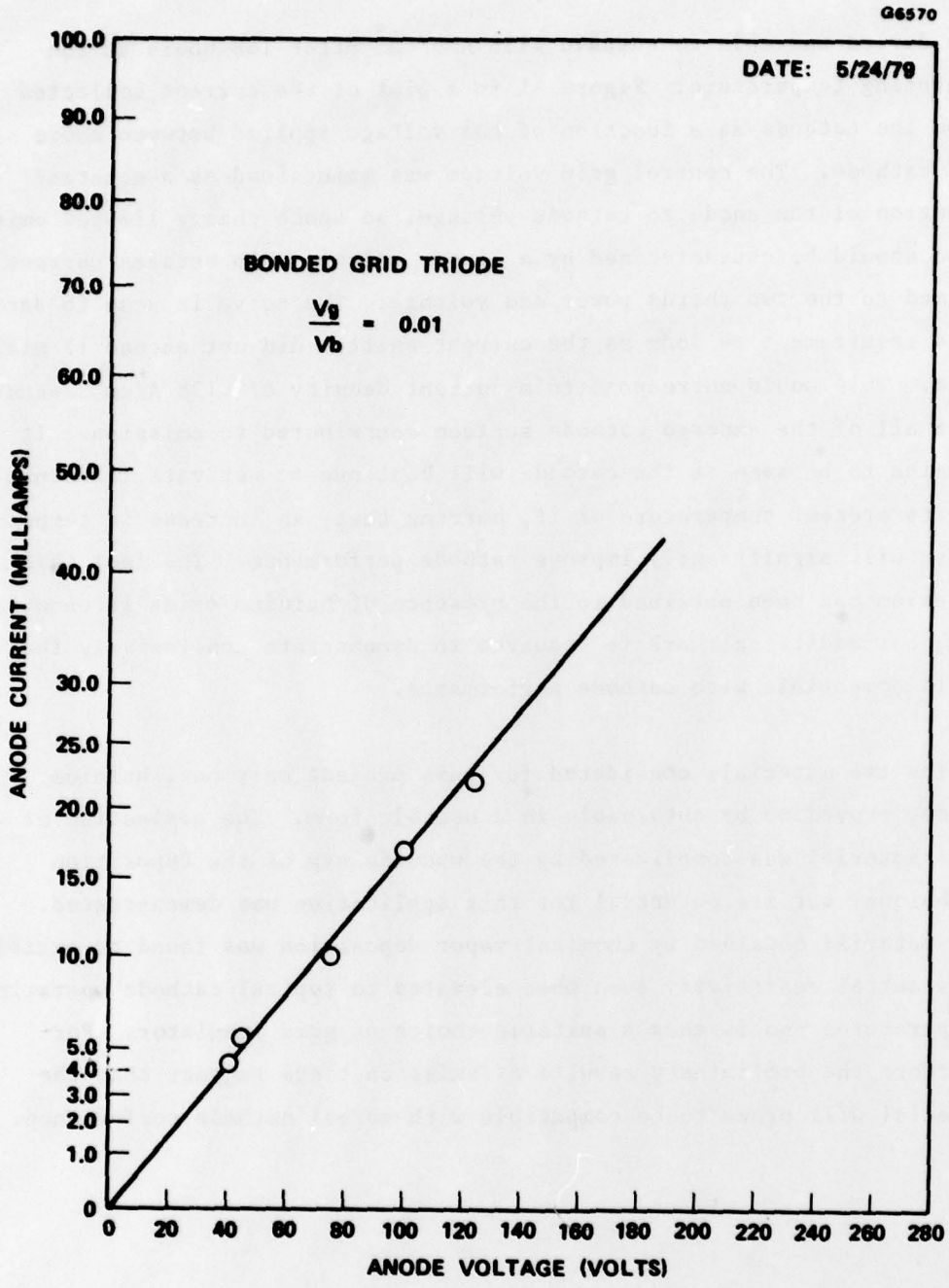

Figure 41 Cathode activation.

SECTION 6
BONDED GRID CHARACTERISTICS

In addition to determining the emission obtainable from the bonded grid assembly, the ability of the grid to control that emission was evaluated by measuring the grid characteristics of the triode device shown in Figure 40 and comparing these data with corresponding values predicted with the aid of a computer program written by W. B Herrmannsfeldt.[4] The close agreement observed between theory and practice suggests that the fabrication of higher mu devices is primarily contingent upon the realization of finer detail in the grid pattern.

The major concern in selecting a grid pattern for this project was to insure that it could be fabricated successfully. Consequently a rather coarse pattern with relatively high screening was used, this structure being most readily produced. Unfortunately such a structure does not provide the most impressive grid control, but as the fabrication techniques evolve it is expected that finer resolution of the grid pattern will allow improved grid characteristics. The success of the present device is thus best measured relative to the theoretical projections for its particular grid pattern rather than relative to any absolute grid voltage requirements which might be deemed desirable. Since the device fabricated here performs as expected, virtually any reasonable set of grid characteristics can be achieved by suitable refinement of the grid pattern.

The predicted and the measured cathode current of the triode depicted in Figure 40 are shown in Figure 42. The cathode current is plotted as a function of the grid voltage applied to the device with the anode voltage maintained at 40 volts. This anode voltage was selected in order that the cathode current required not exceed the capabilities of the cathode. The magnitude of the grid voltage was not allowed to exceed 2.5 volts so as to avoid any unnecessary hazard to the device.

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4. William B. Herrmannsfeldt, "Electron Trajectory Program", Prepared for the U.S. Atomic Energy Commission Under Contract AT (04-3) -515 (9/73).

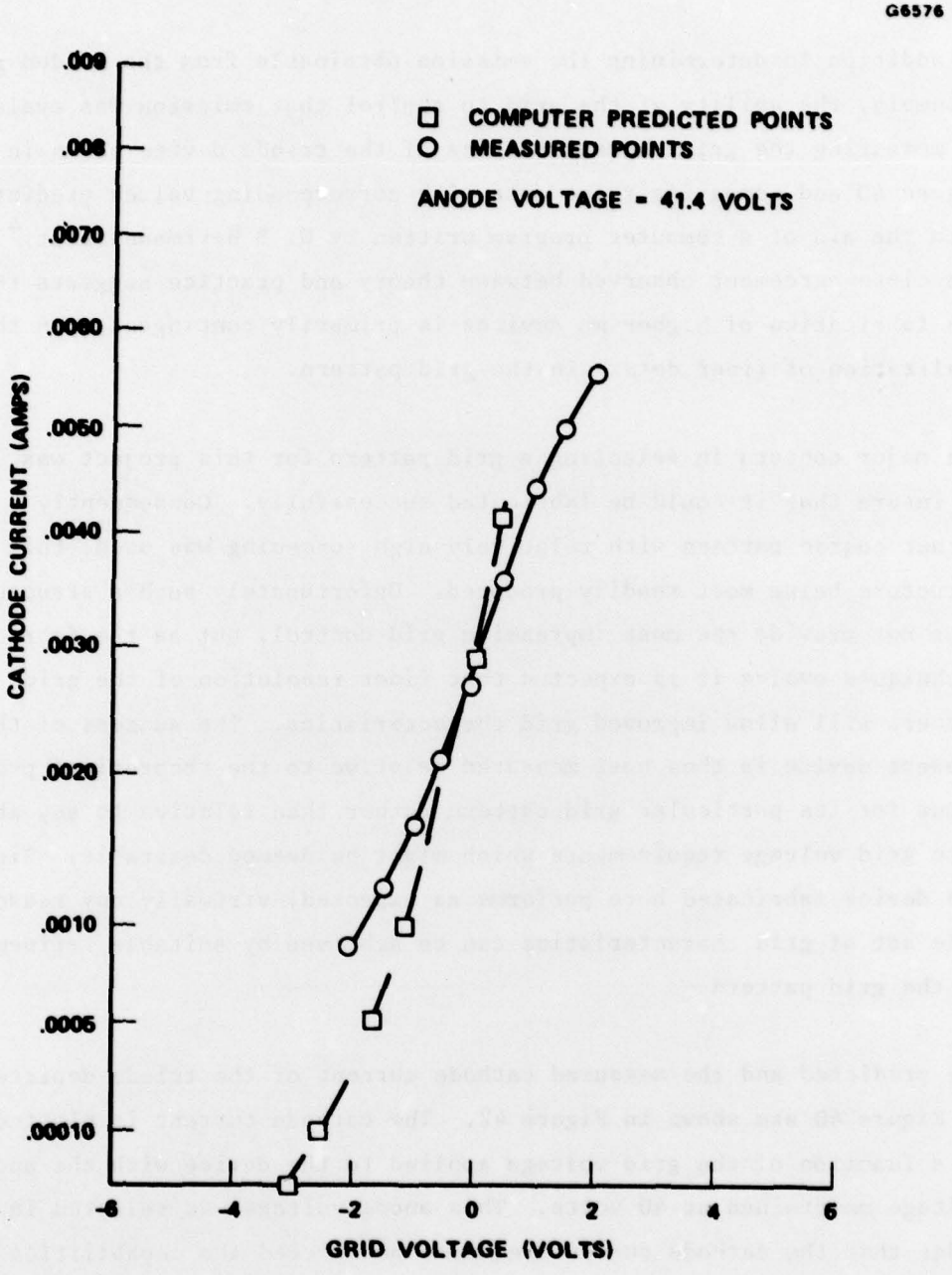

Figure 42 Bonded grid triode grid characteristics.

The actual grid characteristic is seen to agree rather well with that predicted by the computer program, except that in extrapolating the data to determine cut off, the predicted cutoff voltage is found to be too optimistic. This discrepancy remains to be investigated, but the results at hand are encouraging.

In order to extrapolate beyond the information displayed in Figure 42 and infer something of the general usefulness of the bonded grid assembly, it is necessary to recognize that a grid's influence upon cathode emission is moderated by the presence of additional electrodes in the triode, particularly the anode. A first order expression relating the element potentials and cathode current of a triode is as follows:

$$I_K = G\left(V_g + \frac{V_b}{\mu}\right)^{3/2}$$

where

I_K = cathode current
V_g = grid to cathode voltage
V_b = anode to cathode voltage
μ = constant relating to electrode geometry
G = constant relating to electrode geometry

The cut off voltage, that is to say that grid voltage for which cathode current falls to zero, is seen to be a function of both the device's geometry and the anode voltage applied. Similarly the grid voltage required to sustain a given amount of cathode current depends upon both geometrical factors and the anode voltage. Knowledge about the characteristics of a particular triode containing a bonded grid cannot be immediately generalized to describe the behavior of bonded grids under other circumstances, but the fact of agreement between the predicted and actual characteristics tends to verify the design approach

which has been used. Changes to the existing design can be proposed with confidence that the desired results will be achieved.

Basically, two parameters are available to the designer of an electron gun's grid. Following a common practice,[5] the grid's position relative to the cathode is dependent only upon the desired cathode current density and the desired operating voltage for the grid. Similarly, the anode's position can be chosen based upon its desired operating voltage and the cathode current density. With the grid position, anode position, and anode voltage established, the size of the openings in the grid can be adjusted to give the desired cut-off voltage. The first step, picking the grid position or determining the required grid drive voltage if the position is defined, can generally be accomplished with great confidence using only the relationship for current flow between parallel plates:

$$V_g = 5.69 \times 10^3 J_k^{2/3} X_g^{4/3}$$

where

J_k = Cathode current density
X_g = Distance separating grid and cathode
V_g = Control grid to cathode voltage

This relationship can be assumed because the anode is to be positioned in such a fashion as to be consistent with the prescribed grid voltage and current density. The second step, determining the appropriate openings in the grid, is more difficult, as the grid potential cannot be assumed to conform to a known potential distribution during cut off conditions.

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5. H.J. Wolkstein, "Design Considerations for Grid Controlled Electron Guns for Pulsed Traveling-Wave Tubes," RCA Review (9/60).

In the case of the bonded grid assembly prepared for these tests the grid was positioned approximately 0.0018" from the cathode and the anode was 0.060" from the cathode. These dimensions were selected primarily as matters of convenience for fabrication and assembly. With these spacings, parallel plate triode theory predicts the current density at the cathode to be 0.5 amps/cm^2 if the grid is maintained at 2.72 volts and the anode at 292 volts. With only 60 percent of a 0.2012" diameter cathode available for emission, this amounts to 0.062 amps of cathode current to the anode. Reducing the anode voltage to 40 volts and the grid voltage proportionately to 0.373 volts, as was done for testing, should result in 0.0031 amps of cathode current. It is possible to normalize the grid characteristic curves with respect to the anode voltage as has been done in Figure 43 where $J_k/V_A^{3/2}$ is plotted against V_g/V_a. Both measured and predicted values are plotted. In addition, a curve representing a simple parallel plate diode with 0.0018" spacing is plotted. Near the design perveance density ($J_k/V_a^{3/2} = 100.2 \times 10^{-6}$) the curves representing the diode and triode are nearly identical. In this region there is also particularly good agreement between the predicted and actual behavior of the device.

As the grid voltage is reduced to inhibit cathode emission the measured response diverges from that which was anticipated, but the results are in close enough agreement to conclude that conventional analysis techniques are adequate for preliminary evaluation of the bonded grid. The observed discrepancy may be the result of emission from the control grid, since applying a negative grid voltage increases the accelerating field at the grid surface and thus enhances emission from this area. If this proves to be the problem, encapsulation of the grid is proposed to eliminate grid emission, but of more immediate importance is the demonstration that the bonded grid can ultimately be expected to operate with the modest grid voltages which have been predicted.

A typical traveling wave tube application for the bonded grid might require that the average cathode current density be on the order of

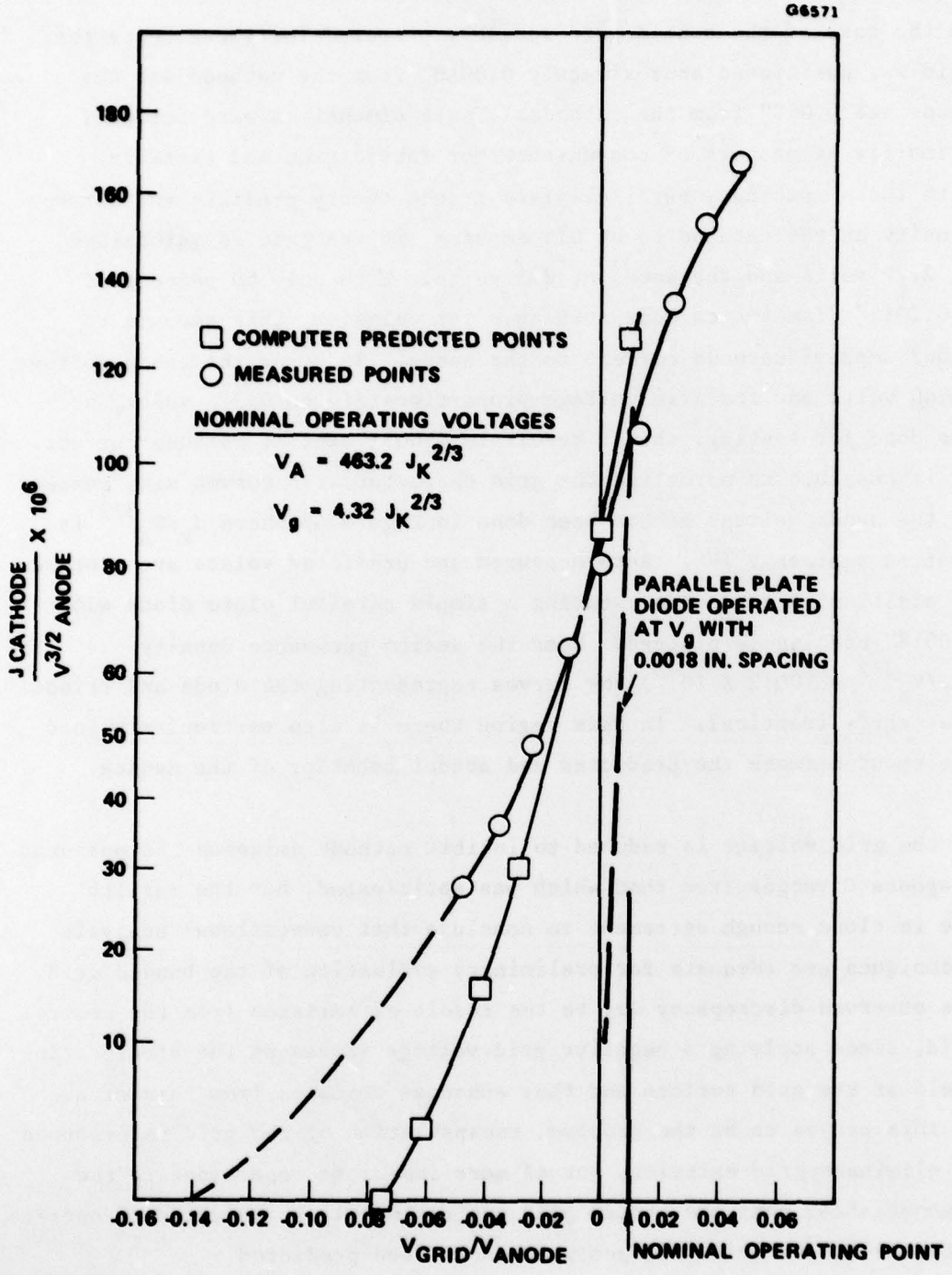

Figure 43 Bonded grid triode grid characteristics.

1.0 Amps/cm^2 and the anode, or accelerating voltage, be several kilovolts. Figure 44 shows the grid voltage required as a function of the current density for the bonded grid triode of Figure 40. The curve of Figure 44 is obtained by extrapolating from the data of Figure 43 and is predicated upon the assumption of space charge limited current in parallel plate geometry. Since the grid is placed so near the cathode in electron guns, the grid voltage required to sustain a particular current density is nearly independent of other geometrical factors in the gun, assuming only that the anode is positioned according to the customary criteria previously mentioned. For this reason a direct comparison of the bonded grid and conventional grids is possible, and typical values of grid drive for several traveling wave tubes are also plotted in Figure 44. The bonded grid is seen to offer the possibility of greatly reduced operating voltages.

Extrapolation of the data concerning cut-off voltage requirements is much more difficult because the effect of the anode depends not only upon the nominal cathode current density, but also upon the anode voltage which has been selected. For a particular cathode current density it can be shown that the cut-off voltage required will increase roughly as the one-fourth power of the anode voltage. In Figure 45 the cut-off voltages of several shadow gridded electron guns are normalized with respect to the one-fourth power of the ratio of their anode voltages to that of the present bonded grid triode when operated with the same cathode current density. The normalized cut-off voltage is plotted with respect to the nominal cathode current density obtained with full grid drive applied. This normalization technique results in a plot of the actual cut-off voltage required for the bonded grid triode, but reduces the value indicated for devices operated with higher anode voltages and allows a direct comparison of the bonded grid with conventional grids. The accuracy of the comparison is extremely limited, but the bonded grid cut-off voltage is seen to be comparable with that of conventional grids under similar conditions.

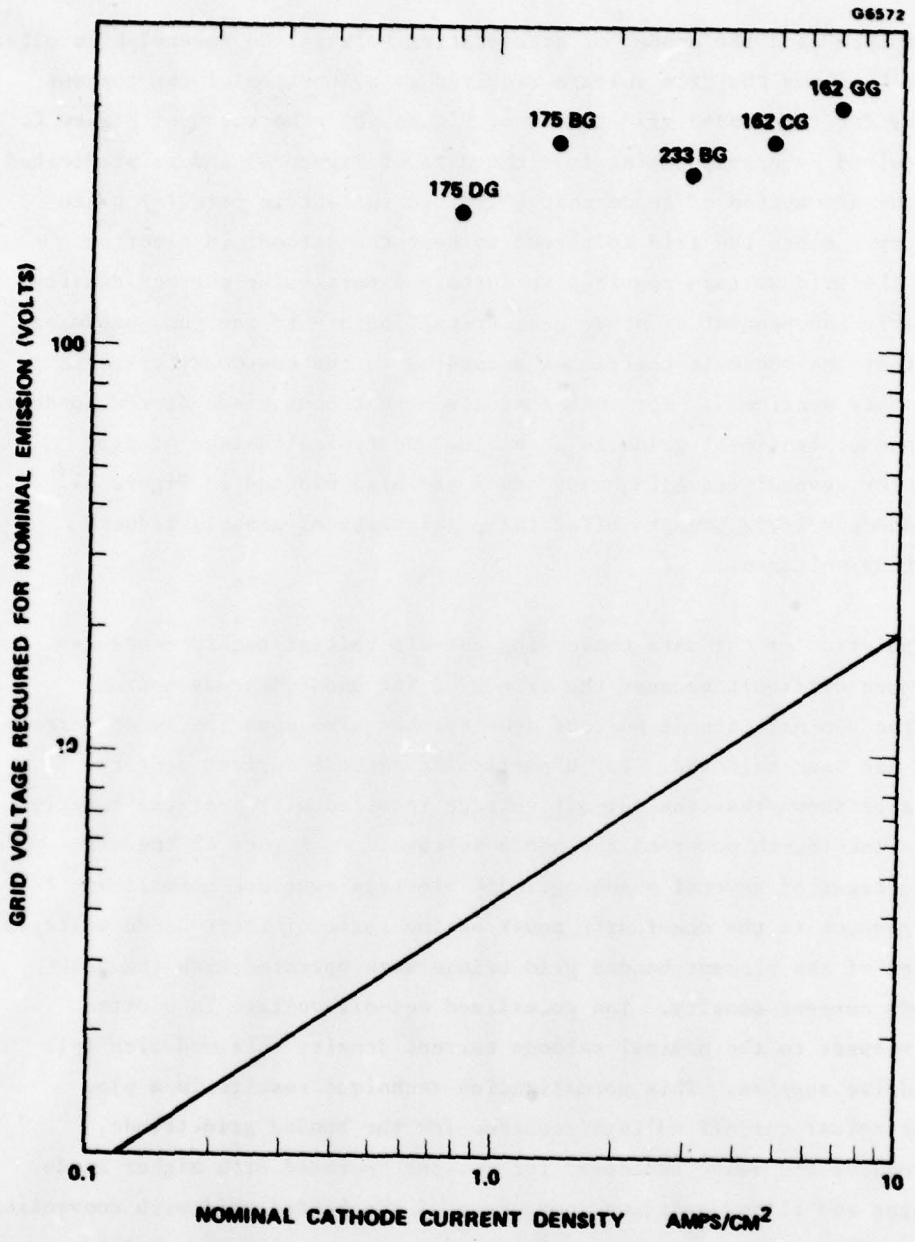

Figure 44 Bonded grid drive voltages compared to those required by typical shadow gridded electron guns.

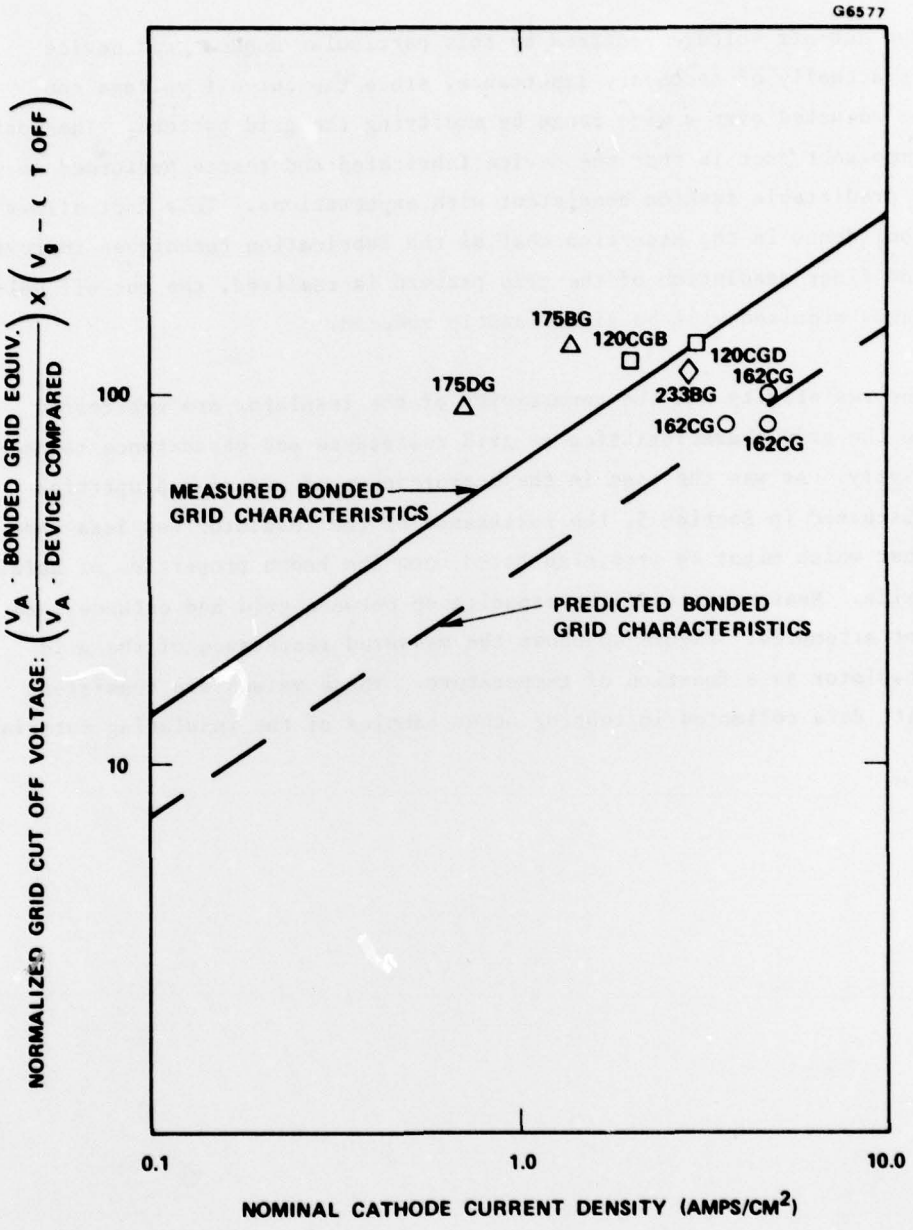

Figure 45 Grid cut off voltage normalized to equivalent anode voltage for comparison of bonded grid to typical shadow grids.

The cut-off voltage required by this particular bonded grid device is actually of secondary importance, since the cut-off voltage can be adjusted over a wide range by modifying the grid pattern. The most important fact is that the device fabricated and tested performed in a predictable fashion consistent with expectations. This fact allows confidence in the assertion that as the fabrication techniques improve and finer resolution of the grid pattern is realized, the cut-off voltages required will be significantly reduced.

The resistivity and the permitivity of the insulator are manifested in the grid characteristics as grid resistance and capacitance respectively. As was the case in the measurements of material properties discussed in Section 5, the resistance of the insulator was less than that which might be predicted based upon the known properties of hafnium oxide. Measurements of the capacitance between grid and cathode were not attempted. Figure 46 shows the measured resistance of the grid insulator as a function of temperature. These values are consistent with data collected in testing other samples of the insulating material.

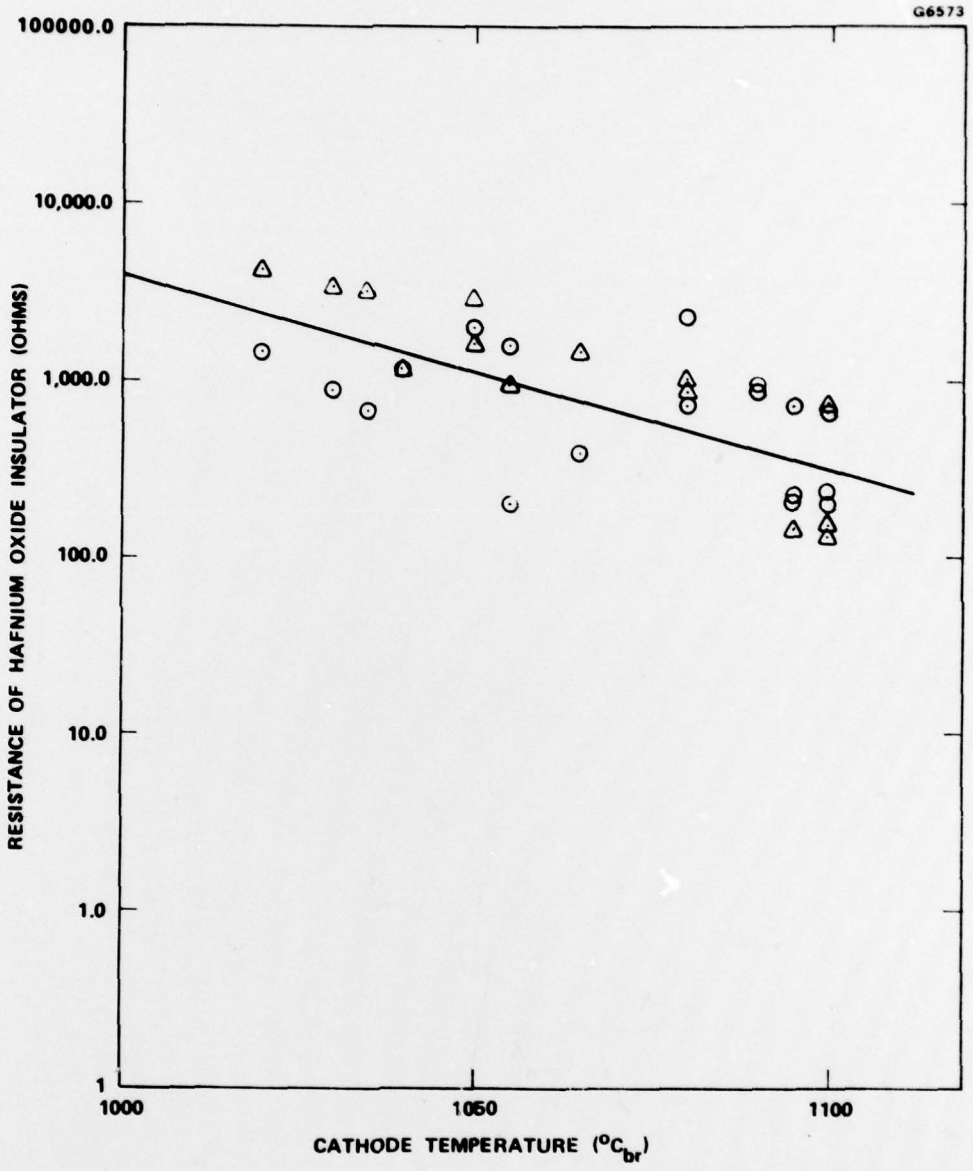

Figure 46 Bonded grid insulator resistance.

SECTION 7
CONCLUSIONS AND RECOMMENDATION

At this stage of development the bonded grid appears to be a promising prospect for the eventual extension of traveling wave tube technology. The successful fabrication and testing of such a device, albeit with limited capabilities, demonstrates the practicality of the manufacturing techniques outlined in this report as well as the potential usefulness of the device itself. Considerable work remains, however, if the many advantages of a bonded grid are to be realized. Subsequent efforts should commence with the optimization of chemical vapor deposition techniques and detailed, comprehensive material studies.

The results obtained in this program show hafnium oxide to be a very promising candidate for use as the insulator in a bonded grid assembly. The material was able to withstand repeated excursions to the expected operating temperatures, and it afforded sufficient isolation between the grid and cathode. Some question remains as to the compatibility of this material with the cathode, however, and the deposition technique needs considerable refinement in order that superior coatings be achieved consistantly. The remaining fabrication techniques were found to be quite effective in producing grids on a flat surface. The mechanical polishing employed to remove excess material would not be well-suited to more complex geometries, however, so some modification of the process would ultimately be desirable if the bonded grid is to find application in convergent flow electron guns. At this point, the more fundamental questions of material properties and cathode compatibility are of paramount importance.

It is recommended that this work continue with the immediate objectives being the final determination of the optimum insulating material

and the development of a more certain means of obtaining that material. Efforts to perfect the manufacturing methods should be postponed in favor of conducting the more fundamental studies necessary to producing and evaluating the insulator. Simple experiments unencumbered by the complexities of creating the intricate grid structure would be best suited to provide final answers to the several key questions which still remain unresolved concerning the selection of an insulator. The results obtained to date are quite encouraging; but, if their full potential is to be realized, this continued effort is essential.